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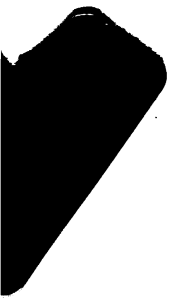

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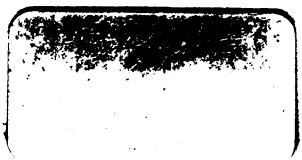

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THE

METALLOGRAPHIST

A Quarterly Publication devoted to the Study of Metals, with Special Reference to their Physics and Microstructure, their Industrial Treatment and Applications.

Edited by ALBERT SAUVEUR.

VOL. III.

1900.

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CONTENTS.

JANUARY, 1900.

New Reagents for the Micrographic Study of Carburized Irons . . . *F. Osmond* and *G. Cartaud* . 1
On Antimonial Alloys for Axle Bearings . . . . . *H. Behrens* and *H. Baucke* . 4
The Constitution of Steel . . . . . *E. D. Campbell* 13
The Present Position of the Solution Theory of Carburized Iron . . . *A. Stansfield* 24
Steels for Magnets . . . . . *F. Osmond* 35
On the Changes of State in Iron and Steel . . . . . *H. Le Chatelier* 38
Colors of Heated Steel Corresponding to Different Degrees of Temperatures . . . . . *Maunsel White* and *F. W. Taylor* 41
The Color-Names for High Temperatures . . . . . *Henry M. Howe* 43
On the Position of the Points of Magnetic Transformation of Nickel Steels . . . . . *L. Dumas* 48
The Microstructure of Steel 52
Constitution of Metallic Alloys 52
Practical Microscopic Analysis for Use in the Steel Industries . . . 64
METALLOGRAPHIC NOTES 83
Léon Gages on Microscopical Metallography. — S. Moutou on Microscopical Metallography. — The Crystalline Structure of Iron and Steel. — The National Physical Laboratory. — Metallography and the Iron and Steel Institute. — The Development of Metallography. — Importance of Metallographic Methods.
CORRESPONDENCE 88
The Critical Ranges in Iron and Steel.

APRIL, 1900.

Henry Clifton Sorby . . . . . FRONTISPIECE
Henry Clifton Sorby, Biographical Sketch 89
The Crystalline Structure of Metals. *J. A. Ewing* and *Walter Rosenhain* 94
The Effect of Heat-Treatment upon the Physical Properties and the Microstructure of Medium-Carbon Steel . . . . . *R. G. Morse* 130
Some Causes of Excessive Heating in Bearing Metals . . . . . *Robert Job* 145
The Dilatation of Iron and Steel at High Temperature . . . . . *H. Le Chatelier* 152
The Microstructure of Coke and Charcoal Pig Irons . . . . . *Albert Sauveur* 154

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The Effect of Annealing on the Magnetic Properties of Steel Sheets 157
Measure of the Abnormal Variations of Length and Temperature taking place in Iron and Steel during the Recalescence 162
The Strains in Completely Hardened Steel 163
Judging Pig Iron by its Microstructure 165
Some Modern Theories on the Cause of Hardening in Steel . . . 165
The Heat-Treatment and Microstructure of Steel 168
Rail Steel: Its Chemistry and Heat-Treatment 173
METALLOGRAPHIC NOTES 174
Phosphides and Sulphides under the Microscope. — The Crystalline Structure of Metals. — The Measurement of High Temperatures. — Metallography and the Steel Industry. — Measurement of Hysteresis of Iron.

JULY, 1900.

Adolf Martens . . . . . FRONTISPIECE
Adolf Martens, Biographical Sketch 177
On the Crystallography of Iron . . . *F. Osmond* 181
Practical Metallography . . . . . *J. E. Stead* 220
Microstructure of Steel Quenched from the Melting-Point 244
Soft Steel 247
The Progress of Metallography 247
METALLOGRAPHIC NOTES 247
Practical Applications of Metallography.—The Allotropic Theory of the Hardening of Steel. — Phosphides and Sulphides under the Microscope. — Metallography at the Technical Schools. — Metallography at the Watertown Arsenal. — Columbia University Lectures. — Professor Samson Jordan. — Erratum.

OCTOBER, 1900.

Floris Osmond . . . . . FRONTISPIECE
Floris Osmond, Biographical Sketch 255
Note on the Structure of Phosphorettic Pig Metals . . . . . *J. E. Stead* 261
The Internal Architecture of Steel . *J. O. Arnold* 267
On the Crystallography of Iron . . . *F. Osmond* 275
Application of the Law of Phases to Alloys and to Rocks . . . . . *H. Le Chatelier* 290
Iron and Steel from the Point of View of the "Phase-Doctrine" . *Bakhuis Roozeboom* . . . 293
The Present Position of the Solution Theory of Carburized Iron . . . *A. Stansfield* 300
The Microscopic Study of Metals . . *W. M. Stine* 314
On Mysterious Fractures of Steel Shafts 320
The Physics of Steel 330
CORRESPONDENCE 338
INDEX 340

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Contributors to "The Metallographist."

H. BEHRENS, Professor at the Polytechnic School, Delft, Holland.

T. A. BRINELL, Chief Engineer, Fagersta Bruks Aktiebolag, Fagersta, Westanfors, Sweden.

G. CHARPY, *Docteur ès sciences*, Engineer, Forges de Chatillon et Commentry, Montluçon, France.

J. A. EWING, F.R.S., Professor of Mechanism and Applied Mechanics, University of Cambridge, Cambridge, England.

G. GUILLEMIN, Metallurgical Engineer, Paris, France.

H. M. HOWE, A.M., S.B., Bessemer Medallist, Professor of Metallurgy, Columbia University, New York, U.S.A.

H. LE CHATELIER, Chief Engineer of Mines, Professor of Chemistry, Collège de France, Paris, France.

A. MARTENS, Director of the Royal Mechanico-Technical Experimental Station, Charlottenburg, Germany.

F. OSMOND, Engineer, Paris, France.

Sir W. ROBERTS-AUSTEN, C.B., F.R.S., Assoc. R.S.M., F.I.C., F.C.S., President Iron and Steel Institute, Professor of Metallurgy, Royal College of Science; Chemist of the Mint, London, England.

J. E. STEAD, F.C.S., F.I.C., Analytical Chemist and Metallurgist, Middlesborough, England.

D. TSCHERNOFF, Professor of Metallurgy, Imperial Academy of Artillery, St. Petersburg, Russia.

H. WEDDING, Ph.D., *Geheimer Bergrat*, Bessemer Medallist, Berlin, Germany.

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A QUARTERLY PUBLICATION DEVOTED TO THE STUDY OF METALS, WITH SPECIAL REFERENCE TO THEIR PHYSICS AND MICROSTRUCTURE, THEIR INDUSTRIAL TREATMENT AND APPLICATIONS.

Vol. III. JANUARY, 1900. No. 1.

NEW REAGENTS FOR THE MICROGRAPHIC STUDY OF CARBURIZED IRONS.*

By F. OSMOND and G. CARTAUD.

ONE of us has recommended for the micrographic study of carburized iron the combined etching and polishing method (*polissage-attaque*), i.e. the rubbing of a section previously polished upon a piece of parchment covered with some aqueous extract of liquorice root, with the addition of precipitated calcium sulphate. It is by this method that he obtained his best preparations.

Liquorice root, however, like all vegetal products, varies greatly according to its origin, the climatic conditions, its state of conservation, etc., resulting in extracts sometimes too active, more often almost passive, the activity of one extract, moreover, varying with its age. It was, therefore, very desirable, although retaining the principle of the combined method, to replace the extract of liquorice root by a reagent of more constant strength.

It first occurred to us that ammoniacal glycyrrhizin, a substance industrially extracted from liquorice root, might contain the useful principle, but its action upon carburized irons was found to be hardly perceptible.

* Received December 16, 1899.

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We then decided to investigate systematically, from our special point of view, the part played by the various chemical functions, either isolated or united in one molecule.

Starting from the benzene nucleus, we studied successively the phenol, aldehydic and acid functions isolated, and then the acid functions united to one or more phenol functions.

The following compounds were tried:

Ordinary phenol . . .	C_6H_5OH
Benzoic aldehyde . . .	C_6H_5COH
Benzoic acid	$C_6H_5CO_2H$
Salicylic acid	$C_6H_4(OH)(CO_2H)$
Gallic acid	$C_6H_2(OH)_3(CO_2H)$

The phenol and aldehydic functions did not have any action upon the samples, even when assisted by friction. On the contrary, the acid function acts in every instance and does not require any rubbing.

We were led, therefore, to look for some compounds in which, when normal, the acid function is neutralized and progressively set free by rubbing the specimen.

The ammoniacal salts in general appear to fill these requirements. The chloride, oxalate and nitrate of ammonium were tried.

The action of the chloride is too weak; the oxalate might be used for the preparation of samples of unhardened steel, but it does not resolve martensite.

A dilute solution of nitrate of ammonium (two parts in weight of the crystallized salt to 100 parts of water) yields good results. A piece of parchment spread tightly over a smooth board is soaked with this solution, and the polished surface of the specimen is rubbed upon it until sufficiently etched. It is not necessary to add any sulphate of calcium. When the parchment becomes dry a little water is poured over it.

The results are exactly those produced by the extract of liquorice.

The various constituents are identified as follows:

1. Pearlyte by the unequal depth of etching of its two components, and sorbite by its coloration varying from light yellow to dark brown.

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2. Troostite by its yellow, brown or blue bands merging into each other.

3. Martensite by its characteristic needles, which are the better defined the less carbon in the metal.

4. Ferrite by its division into grains and the heterogeneous appearance of these grains.

Austenite and especially cementite remain unacted upon.

It must not be supposed, however, that this combined etching and polishing method by means of nitrate of ammonium is free from all difficulty, nor must it be expected to succeed at the first trial. The reagent has a tendency to form a film of oxide which must be removed by rubbing as soon as it is formed, and success depends upon a complex set of conditions (concentration of the solution, pressure, speed, quality of the parchment, etc.), several of which belong to the personal equation and could not be stated accurately. Every one must make an apprenticeship, but once the routine of the method acquired, martensite may be resolved in 400 frictions, back and forth, that is in less than three minutes of effective work.

A satisfactory means of distinguishing austenite from martensite was still to be found. It can be accomplished by immersing the metal in a solution of chloride of ammonium, a 10 per cent solution for instance, while it is connected with the positive pole of a bichromate cell, the negative pole consisting of a piece of platinum foil, or of lead, or iron foil. The specimen is examined every 10 seconds until the etching is sufficiently deep. A very dilute solution of hydrochloric acid may also be used, with or without the assistance of an electrical current, but the use of a battery is always desirable as it increases the regularity of the chemical action. In both cases martensite is first colored, successively yellow, brown and black.

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ON ANTIMONIAL ALLOYS FOR AXLE BEARINGS.*

By H. BEHRENS and H. BAUCKE.

IN 1897 several cushions of Babbit metal (containing 82 per cent of tin, 9 per cent of antimony, and 9 per cent of copper) were put aside by the manager of the "Holl. Spoorweg My." for a thorough chemical and microscopical examination with a view of ascertaining the causes leading to hot boxes. The microscopical and mechanical properties were determined by Professor Behrens, while the chemical examination was undertaken by Mr. Baucke.

1. *Structure of Antimonial Alloys for Bearings.*—The principal microscopical features of alloys containing tin, antimony and copper, were already known (H. Behrens, *Das mikroskopische Gefüge von Metallen und Legierungen*, 1894, page 60). Alloys of this kind had been examined, containing from 7.5 to 15 per cent of tin and from 5.5 to 11 per cent of copper, and had been found, when slowly cooled from a melting heat, to split up into rectangular crystals (probably cuboidal rhombohedrons) of an alloy of tin and antimony and into a whitish bronze, forming radial clusters of brittle rods, composed of hexagonal plates. The mother-liquor or matrix was found to be nearly pure tin. The cuboidal crystals were evidently identical to those of Britannia metal (9 parts of tin, 1 part of antimony), the rods of whitish bronze identical to those of an alloy, composed of 9 parts of tin and 1 part of copper. It was at the time suggested that the axle was supported by the smooth cubes, while the hard rods of bronze prevented the axle from being contaminated by the soft and adhesive tin. On the whole the observations and experiments made in the last years agree with the views put forth in 1893; in some details, however, where a better insight has been gained, these views must be modified.

2. *Properties of the Morphological Constituents.* — The bronze-needles are the hardest among the constituents (hard-

* Received December 28, 1899.

The substance of this paper was published in *Baumaterialkunde*, Vol. IV, Parts 6 and 7. It was re-written and translated into English by Professor Behrens for the present number of *The Metallographist.*—ED.

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ness = 2.5, lead = 1, copper = 3), while they also show the highest melting point and accordingly are the first to crystallize. In alloys of copper, containing from 85 to 95 per cent of tin, they are always present, even in chilled castings, becoming more numerous and of greater size (up to 2 mm. in length) when the metal is slowly cooled. On well prepared slides they have a light yellow tint, pointing to about 30 per cent of copper, but it is not at all easy to obtain the necessary finish. Generally the specimens are full of blackish striae, caused by the crumbling of the yellow needles. This excessive brittleness of the bronze needles cannot be made to accord with the supposition that they form a trussing in the axle-box. It is also unlikely that they help in keeping the axle clean, acting as scrapers, because they will crumble under a loaded axle even more rapidly then by rubbing on a whetstone. The microscopical fragments of whitish bronze are angular, softer than iron, but of sufficient hardness to scratch tin. From the crushing of the bronze needles and subsequent grinding of the tin a *relief* must be formed on the bearing surfaces of the cushion blocks, somewhat similiar to that produced by etching. It will be seen that this is a circumstance of great consequence with regard to the efficiency of lubrication, the oil being *spread evenly* over the bearing surface and *kept* in this condition, even when the axle is heavily loaded and running with great speed. The cuboidal crystals, composed of tin and antimony, have a lower degree of hardness (2.1, tin = 1.7) than the bronze-needles, and a lower melting point. They are sufficiently harder than tin to produce a sensible relief by rubbing with fine emery powder. The cubes are neither brittle nor fissile; by hammering they may be flattened to twice their original size. Alloys low in antimony, and alloys that are rapidly cooled yield crystals of small size (0.01 mm.). With less than 4 per cent of antimony no crystals were obtained, with more than 40 per cent of antimony large rods and plates did appear in place of cuboidal crystals. Large cuboids (0.4 to 0.6 mm.), such as are formed in slowly cooled cushions with 10 to 15 per cent of antimony, are sometimes pierced by several needles of bronze, more brittle even than those of the matrix mass, leaving deep furrows and rents, whereby the cuboid is weakened.

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The crystalline constituents of Babbit metal are bound together by a ground-mass or mother liquor, principally of tin, containing a percentage of antimony and copper which is the greater the more rapidly the metal is cooled. In chilled metal the hardness rises to 2, by cooling very slowly the hardness of the matrix can be lowered to 1.6. In cushion blocks of normal condition it is 1.7 to 1.8.

3. *Melting of Babbit Metal.* — Babbit metal is made without any difficulty. The copper is fused under a layer of charcoal, then the same weight of tin is added in two or three portions, each with a light stirring, then the antimony, and finally, with thorough stirring, the rest of the tin. After the last addition the heat is reduced so far that casting may follow without delay. The temperature for casting is near the melting point of zinc, higher than that of lead.

When such an alloy is fused the matrix is liquified first, at a temperature nearly coinciding with the melting point of tin. From a comparison of the specific gravities — tin 7.2, copper 8.95, antimony 6.7 — we should be led to expect a layer of cuboidal crystals, composed of tin and antimony, segregating at the surface of the metallic bath. This expectation is not realized. The crystalline constituents sink to the bottom of the melting pot, accumulating there into a pasty mass, which requires a considerable amount of heat and stirring to be liquified or rather to dissolve in the molten tin. During this interval oxidation is inevitable. Hard grains and lumps of oxides are formed, and in consequence of their high specific gravity (6.5 to 6.7) and the repeated stirrings, are worked into the metal. The contact of one or more of these hard lumps with a running axle will lead to deterioration of the axle and to violent friction, and this must result in heating and finally in liquefaction of the bearing. It is far better to seperate the pasty mass from the tin, to liquify it under charcoal at a low red heat and then to stir in the tin previously removed. Some antimony is lost by volatilization.

4. *Casting in Hot and Cold Moulds.* — It is asserted by engineers that cushion blocks of "white metal" will be of good quality if they are cast around a core having the temperature of boiling water, while, on the contrary, castings of the same

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metal made with a red-hot core or a cold core will cause hot boxes. Modification of structure must here come into play, and to investigate the influence of the core, castings on a reduced scale were made. First, a red-hot pin was inserted in a mould filled with Babbit metal and kept hot, so that solidification required an interval of 10 minutes. The pin having been knocked out, the front of the block was ground and etched with strong hydrochloric acid. Round the central hole the cuboids measured 0.5 mm., the bronze needles (excessively brittle) 2 mm.

Another block of the same metal was cast around a tubular core cooled by running water. At a distance rudimentary cuboids were seen, while around the core the metal was nearly amorphous, showing only a few minute rods of bronze. Plates of chilled metal were prepared by casting on a slate and cooling the liquid metal under a block of cold iron. By chilling, Babbit metal is made very fine grained, sonorous and very smooth when filed or turned.

A third block, cast around a core heated to 100° C., showed a normal structure, with numerous cuboids measuring 0.2 to 0.25 mm. Hardness of the ground mass = 1.7.

5. *Structure of Sound and of Defective (Heated) Bearings.* — Samples cut from bearings tried by long service under railway cars showed a normal structure: the crystalline constituents well developed, and of medium size. The greatest cuboids measured 0.24 mm., they were accompanied by a great number of small ones, from 0.03 to 0.08 mm. Samples taken from cushions which had become hot exhibited two different types of structure, one, characterized by cuboids measuring 0.4 to 0.6 mm., separated by cavities and grains of oxides, the other characterized by a few rudimentary crystals in a compact hard matrix (hardness 1.9 to 2). A cushion block of the last named type, from which liquid metal had trickled, proved very instructive. The droppings consisted of tin, with a slight admixture of copper and antimony in the shape of small crystals carried along by the liquid tin. On a transverse section of the cushion a white shining ring was seen, about 2.5 mm. broad, encircling the axle. It was composed of great cuboids, packed closely around the axle, while at a distance of 10 mm. the cuboids were small and scanty, and at a distance

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of 30 mm. absent altogether. Here the metal had evidently been chilled, and it was to be surmised that the ring of great cuboids must be due to *recrystallization*, caused by local heating.

6. *Recrystallization of Chilled Babbit Metal.* — A plate of chilled metal was made angular by filing the edges, then heated over a small flame at an edge or corner till the sharp angles at this point were seen to shrink and get rounded. After grinding and etching the same structure was found as in the defective cushion described in (5). Numerous large cuboids were found in the immediate vicinity of the heated spot, while farther off the crystals were smaller and scattered, and at a distance of about 8 mm. none appeared. Experiments with polished plates of chilled metal have shown that the limit of recrystallization coincides with the limit of softening (fusion of the ground mass), and this was found to be slightly superior to the melting point of tin.

7. *Experiments on the Behavior of Cushions Varying in Structure.* — For experiments on the running qualities the model blocks described in (4) were used. The block cast around a cooled core was labelled I, the block cast around a red-hot core, II, the block cast at normal temperature (core 100°) was labelled III. They were planed and turned to the same size, so as to fit in a wooden casing, on a mandril of polished steel,* running at a speed of 1600 revolutions per minute. The apparatus was contrived in such a manner, that the pressure of the blocks on the mandril could be varied at pleasure and the temperature observed on a thermometer fitted into the blocks by means of a soft amalgam. In the course of a preliminary trial block I was found very liable to sticking, block III was free from this defect. The mandril had taken a greyish tint, and its polish was somewhat tarnished. By rubbing with filtering paper and a little barium sulphate it was brightened while the paper took up a notable quantity of tin. Evidently, tinning and sticking are nearly allied.

The following table gives for some of the experiments the pressures reduced to kilograms per square centimeter of

* Diameter 15 mm., length of bearing 35 mm.

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the longitudinal section of the mandril, and the mean increase of the temperature after a minute of running.

	0.3 kg.	0.4 kg.	0.6 kg.	1.2 kg.	3.0 kg.	Pressure.
I . .	0.50	0.82	1.12	1.50	3.80	Increase of temperature.
II . .	0.65	1.60	1.72	2.62	4.64	
III . .	0.64	0.64	0.74	0.75	1.64	

The formation of the concentric layers in the defective block, described in (5), is now easily explained. Originally it was a uniform mass of chilled metal. Notwithstanding its hardness and smoothness this will stick to the axle and tin it. As an inevitable consequence heating must have ensued, ending in recrystallization and squeezing out of liquid tin, the newly formed crystals accumulating around the axle.

After the series of experiments with a charge of 1.2 kg. block I presented a surface scarred with irregular grooves and scratches. On block III the rectangular crystals stood out in relief, similar to that produced by etching with hydrochloric acid. They were smooth and bright, the interstices deadened by small scars or dimples. On block II the features exhibited by I and III were found combined.

Running with a charge of 3 kg. made these features more striking yet. On block II many of the great cuboids were deeply scarred by the crumbling of inclosed needles of bronze, several showed cracks and were partly crushed. On block III some cuboids were rounded, and these stood out in a circular groove, as if they were about to be undermined and loosened.

Examination of the metallic sediment from the oil that had been used as lubricant proved that undermining and loosening of the cuboid crystals does indeed occur. Mixed with fine dust (essentially powdered tin) were found: in sediment from block I, shavings, threads and angular fragments; in sediment from block III, spherical and egg-like bodies (0.08 to 0.1 mm.), looking like small drops of mercury; in sediment from block II, spheroids and angular fragments. A few of the spheroids from block III were subjected to microchemical tests, and found to consist of tin with a considerable admixture of antimony.

These observations suffice to explain the slow increase of temperature in block III. The oil was more evenly spread, owing to the peculiar relief developed on the running surface

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of this block, and at the same time a *ball cushion* was formed, whereby a rolling instead of a sliding friction occured. Spheroids similar to those of block III were found in oil from the engines of two steam boats, one running on Babbit, the other on Magnolia metal (77.8 per cent Ph, 16.3 per cent Sb, 5.9 per cent Sn). In oil from bearings of Magnolia metal, poor in cuboid crystals, the spheroids were scarce and small (0.03 to 0.05 mm.). Spheroids were also formed in bearings of aluminium brass, containing hard grey buttons and rosettes (hardness = 3.7), but not in bearings of common brass and of grey cast iron. In this way their origin has been traced to cuboidal or polyhedral and nearly isometrical crystals, scattered in a softer metal and accompanied by smaller crystals of a hard and brittle alloy. In Babbit metal fragments of the brittle rods of bronze act as a grinding powder, undermining and rounding the rectangular crystals of the compound $SbSn_2$ in the same way as pebbles are formed by the grinding action of quartz-grains in the bed of a river. The tin comes in as a soft cement; possibly its powder has also a favorable influence, augmenting the viscosity of the lubricant. Metal of coarse structure (cast in overheated moulds) is not evenly attacked; the majority of the cuboidal crystals, weakened by cracks, are scarred and crushed instead of being rounded and worked up into spheroids. Spheroids must accumulate in a bearing, because of their running in circles, while angular fragments will follow irregular tracks and after a short time be cast out with the surplus of lubricant.

8. *Chemical Examination.* (By Mr. H. Baucke.) — As our knowledge of the composition of Babbit metal from a qualitative point of view may be considered satisfactory, the chemical investigation was confined to the separation of the crystalline constituents and to quantitative analysis of the products. On the other hand it was extended to alloys much richer in antimony.

Alloys of copper and antimony not being present in Babbit metal, it was feasible to work separately with bronzes and with alloys of tin and antimony. For separating the products of liquation advantage was taken of the difference of melting points. When Babbit metal in a pasty, semi-liquid condition is squeezed between hot slabs of iron, *tin* will flow

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out, containing about 3 per cent of copper and antimony, and a hard, brittle cake will be left, consisting principally of the crystals described above.

a. When an alloy of tin with 10 per cent of antimony was subjected to such treatment, after three pressings the remaining cake had an homogeneous appearance. It was clipped at the edges, the central portion was crushed in a heated mortar and sifted. The metallic powder was briskly attacked by concentrated hydrochloric acid at the ordinary temperature. Tin was dissolved, while the fragments of the cuboidal crystals retained their color and brightness. A black slime of antimony was washed away in a stream of water. Analysis of the dried residue gave the following results:

Found	Calculated from the formula $SbSn_2$
Sb 33.7	Sb 33.8
Sn 66.3	Sn 66.2

Mr. Charpy gives the formula SnSb (in *Bulletin de la Société d'Encouragement*, 1898)* without analytical proof. He takes the ground mass for an eutectic alloy, composed of crystalline grains. The mother-liquor is in fact crystalline and contains a small quantity of antimony. Alloys of this kind are considered by v. Bylert (*Zeitschr. f. physik. Chemie*, 8, 348) as solid solutions, the melting points rising with the percentage of antimony.

With 45 per cent of antimony an alloy was formed containing cuboidal crystals, prismatic crystals that seemed to belong to the rhombic system, and a small quantity of granular grey ground mass. With 64 per cent of antimony all the crystals have taken the prismatic form. Pressing between hot plates and subsequent treatment with hydrochloric acid, as above described, left a light grey residue, analyzed with the following result:

Found	Calculated from SnSb
Sn 49.65	Sn 49.63
Sb 50.35	Sb 50.37

Here we have thus before us a second compound of tin and antimony, answering to the formula Sn_2Sb_2.

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* *The Metallographist*, Vol. II (1899), page 34.

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With a higher percentage of antimony the structure is changed once more, laminated crystals of *antimony* appearing between the prismatic ones of the compound Sn_2Sb_2. The presence of a third substance is probable, encasing the prismatic crystals. The difference of melting points has now become too slight to permit separation by means of pressure.

Hydrochloric acid does not attack the metal, and a mixture of this acid with chlorate of potassium gives corroded specimens that are quite useless. Better results were obtained by means of iodine. Polished specimens were put into a bottle containing some dry iodine. After some time, depending upon the temperature of the laboratory, the antimony was stained red, the encasing substance a reddish orange, the prisms, poorest in antimony, a greyish yellow.

b. Alloys of copper and tin.

Alloys of tin with 10 per cent of copper require a peculiar treatment to effect a satisfactory separation by hot pressing. The first squeezing gave a mother liquor, containing 2.9 per cent of copper, and a cake full of crystalline fragments. This being subjected to repeated squeezings, it was found that each time a higher temperature was required to make the cake pasty, and each time the cake and the mother liquor became richer in copper. Afer the fifth squeezing the cake contained 52.77 per cent of copper, after the sixth squeezing between red-hot slabs, it had reached 58 per cent. Evidently each rise of the temperature expelled some tin, thereby adapting the crystalline compound to the new conditions. To obtain definite results it became necessary to simplify the experiment. A big lump of alloy, 720 gr. of tin mixed with 80 gr. of copper, was pressed once as thoroughly as possible. In the cake the majority of the crystals had retained their shape. The central portion was crushed, the metallic powder treated at ordinary temperature with hydrochloric acid of 1.19 sp. gr., washed, and the last traces of mother liquor eliminated by a treatment with strong soda ley and repeated washing. Microscopical examination of the residue showed small hexagonal prisms, often capped by a pyramid. These crystals are not attacked at the ordinary temperature by hydrochloric acid 1.19 sp. gr. nor by nitric acid 1.2 sp. gr. In aqua regia and in hot nitric acid they are dissolved, total solution in the last named solvent

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being only reached after repeated evaporation. Quantitative analysis gave the following results:

Found	Calculated from CuSn
Cu 35.1	Cu 34.9
Sn 64.8	Sn 65.1
99.9	100.0

From this analytical result, combined with the great chemical stability and well developed crystalline form of this metallic substance, the inference may be drawn that in bronzes, containing more than 70 per cent of tin, and in white alloys for axle boxes, containing copper and tin, a chemical compound is formed answering to the formula CuSn. Compounds with a higher percentage of copper are not present.

From fractional crystallization and subsequent analysis Mr. Förster drew the conclusion that in an alloy containing 90 per cent Sn + 10 per cent Cu two different compounds of copper and tin were present. His reasoning will not hold good, inasmuch as the crystals and the mother liquor must necessarily differ in composition, the initial mixture containing more tin than the crystallizing compound CuSn, and the residues prepared by him from the fractions by extraction with hydrochloric acid must also differ in composition, considering that the residue left by the crystalline mass will consist chiefly of crystals of the compound CuSn, while the residue from the decanted fraction will contain crystals of the same kind, mixed with copper, left by the mother liquor proper. Moreover, in specimens of the slowly cooled alloy *only* hexagonal rosettes are seen, composed of minute hexagonal crystals.

THE CONSTITUTION OF STEEL.*

By E. D. CAMPBELL.

IN the interesting paper read at the last meeting† by Professor Arnold and Mr. M'William, the authors say, "Professor Campbell seems in error on one important point,

* Iron and Steel Institute, August 1899 meeting.

† *Journal of the Iron and Steel Institute*, 1899, No. I, page 85. *The Metallographist*, Vol. II, page 305.

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namely, with reference to the alleged non-diffusive power of pure sulphide of iron." I feel, however, that if the different conditions under which the authors' and my own experiments were carried on are considered, it will be seen that there is, after all, nothing in the experiments described that will contradict the statements in my own paper. Although my own experiments were originally undertaken to prove that an undoubted chemical compound can diffuse through steel, I have, myself, never felt satisfied that the passage of sulphide of iron through steel could, in strict sense, be regarded as true diffusion; it has always seemed to me to be a phenomenon rather of filtration through a porous body, than true diffusion. To me the phenomenon of true diffusion in solids involves, first, solid solution of the diffusiate in the entire mass, and intermolecular movement of the diffusiate, whereas filtration involves the passage of the filtrate in liquid form through the capillary spaces between the solid particles of metal, these particles being of very appreciable size. Professor Arnold has himself very ably shown microscopically that sulphide of iron occupies the minute interstitial spaces between the relatively large particles of iron, rather than occurs in solid solution throughout the mass of the metal, and my own experiments have shown, by the tendency of the sulphides to accumulate near the lower part of the bar, that the diffusiate is in liquid form, during its passage through the steel. It would seem, then, to me that, if what I have termed the diffusion of sulphides through steel is, strictly speaking, a phenomenon of capillarity rather than true diffusion, we might justly expect the diffusive power of different substances to be influenced by the same conditions that would affect ordinary filtrations. It is well known that if the cohesion of a liquid is greater than its adhesion to the walls of a porous vessel containing it, the liquid will not enter the pores of the container and filter through it; on the other hand, a liquid may be drawn through a porous body by a vacuum when it would not enter the pores under atmospheric pressure. This is illustrated by the familiar example of the filtration of water and of mercury through a longitudinal block of wood; the water will readily penetrate and filter through, even under ordinary pressure; the mercury will remain on the upper sur-

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face of the wood so long as the pressure above and below are the same, but will be forced through if a vacuum is produced beneath the block. To me, the differences in result on oxysulphide and pure sulphide of iron are due to just this same difference in conditions of experimentation. In several of the experiments made in this laboratory, the plugs used for closing the hole during the diffusions were raised out of position, thus giving evidence of more or less internal pressure, although this pressure does not seem to have been very great. In my own experiments, therefore, only those sulphides whose cohesion was less than the adhesion to the walls of the capillary spaces in the metal, would diffuse or filter through the iron and be absorbed by the porous scale on the exterior, or the asbestos on which the bar rested. The cohesion of pure sulphide of iron seemed to be greater than the capillary attraction of the pores of the steel, and in consequence this substance remained practically unchanged in the bar, when the outside of the bar was at atmospheric pressure, although Professor Arnold and Mr. M'William have shown that the pure sulphide will be drawn through the pores of the steel if the bar is *in vacuo* during the experiment. I feel that the experiment serves rather to confirm than to contradict the conclusions drawn from my original experiments, namely, that steel must be regarded as a porous body, through the pores of which a liquid may be made to filter, provided the cohesion of the liquid is not too great. Oxysulphide of iron is such a mobile liquid, that is, has such low cohesion, that it will enter the pores of the steel and filter through, assisted only by capillary attraction. Professor Arnold has now demonstrated that, although the cohesion of pure sulphide of iron is greater than the capillary attraction of the steel, nevertheless this cohesion may be overcome, and the liquid sulphide be drawn through the pores of the steel, provided the external pressure is sufficiently reduced.

The results obtained by Professor Arnold in his experiments on the diffusion of phosphides seem to me to be of much more significance than those obtained with sulphides. In the case of the phosphide, judging from his microscopical examinations, the phosphorus compound, whatever it may be, seems to be in true solid solution, evenly distributed through

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out the entire mass, and, consequently, to have diffused, in the strict sense of the word, from the inner core into the surrounding jacket. The sulphide, on the other hand, has been drawn by capillary action for a short distance into the interstitial spaces of the steel. This interstitial penetration will not extend far into the jacket, since the penetrating sulphide, instead of existing pure and in relatively unlimited amount as in the experiments conducted here, already occupies the interstitial spaces of the core, so that there would be little tendency for it to change its location, the capillary attraction of the containing interstitial spaces being as great as that of the surrounding jacket.

Now that Professor Arnold seems to have demonstrated by his experiments with phosphorus that diffusion in the strict sense of an undoubted chemical compound can take place in iron, I feel as he does that there is no experimental proof that the diffusing carbon in iron is necessarily in the elemental form. Although in the experiments of Mr. G. P. Royston* it would seem at first glance that the diffusing carbon must be in the elemental form, a consideration of the laws of solution will show that this assumption need not necessarily be made. If we regard the carbon in steel at, say, 850° C. to be present in the form of dissolved carbides or ferro-carbons, and that these dissolved ferro-carbons are more or less ionised, it would seem to me much easier to conceive how carbon ions might migrate from high-carbon into low-carbon steel without losing their chemical relation to iron ions, than it would be for me to understand how we could obtain the same products of solution from a definite chemical compound of iron and carbon that we would obtain from a mere mixture or solution of free uncombined carbon in iron. Before the carbon solution theory can be considered as definitely proved, it will be necessary for the "carbonists" to bring forward some satisfactory explanation of the behavior of solvents toward annealed and hardened steel. Although our experiments are far from complete as yet, and we hope to continue the investigations along the present lines of research, the results so far obtained may serve to throw a little light upon the vexed question of the constitution of steel.

* *Journal of the Iron and Steel Institute*, 1897, No. I, page 166 *et seq.*

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In my discussion of Mr. A. Sauveur's paper on "The Microstructure of Steel and the Current Theories of Hardening,"* I stated the firm belief which I held at that time, and which I still hold, that we are not only justified in depending upon the products of solution of steel for deducing the constitution of the iron and carbon compounds, but we must turn to these products for the desired information in regard to these bodies. In studying the constitution of practically all chemical bodies, whether organic or inorganic, it is assumed that if two compounds under the action of the same solvent and same conditions of solution yield the same or similar products, the bodies dissolved have the same or similar constitution. It is upon this assumption that our present investigations were commenced some eight years ago, and although our results are far from complete, they point towards some rather interesting conclusions, which we hope to be able to verify by means of further work along the lines indicated by the results so far obtained.

As early as 1864 Hahn† obtained from different cast irons a variety of hydro-carbons, a part being gases, belonging to the olefine series (i.e. having the general formula C_nH_{2n}); a part gases of the paraffin series (general formula C_nH_{2n+2}); and a part unidentified liquid hydro-carbons. Later, in 1877, Cloez‡ dissolved a large sample of manganiferous white iron, and recovered part of the carbon by absorbing the gaseous olefines in bromine, thus converting them to the di-brom derivatives, this absorption taking place according to the equation: $C_nH_{2n}+Br_2=C_nH_{2n}Br_2$. In addition to the carbon obtained in the above manner, Cloez obtained also a portion of his carbon as low-boiling liquid paraffins, especially $C_{12}H_{26}$, together with some unidentified liquid hydro-carbons. Both Hahn and Cloez proved qualitatively that the olefines — ethylene (C_2H_4), propylene (C_3H_6), butylene (C_4H_8), and pentylene (C_5H_{10}) and di-butylene (C_8H_{14}) — were present in the gases; and Cloez identified the paraffins from decane ($C_{10}H_{22}$) to hexa-decane ($C_{16}H_{34}$). Both mention that duo-

* *Transactions of the American Institute of Mining Engineers*, Vol. XXVII, page 869 *et seq.*

† Liebig's *Chemical Analysis*, Vol. CXXIX, page 57.

‡ *Comptes Rendus*, Vol. LXXXV, pages x, 3.

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decane ($C_{12}H_{26}$) was the predominant member of the oily paraffins. The work of neither of these chemists was strictly quantitative; and neither attempted to account for the total amount of carbon, or to deduce from their results any generalizations which might lead to a theory concerning the relation of carbon to iron.

The general method which we have employed for studying the products of steel has been to dissolve the steel in hydrochloric acid, pass the gas evolved through bromine in order to convert unsatured hydro-carbons of the general formula C_nH_{2n} into their di-brom derivatives $C_nH_{2n}Br_2$; the gas passing through the bromine being measured, and the carbon existing as gaseous paraffins being determined by explosion and absorption of the carbon dioxide produced. The di-brom derivatives, after proper purification, drying and weighing, are analyzed and fractionally distilled for the purpose of qualitatively identifying the various constituents; although the fractional distillation of the di-brom derivatives has shown the presence of ethylene, propylene, butylene, pentylene, and hexylene di-bromides, and dibutylene tetrabromide, later investigations have convinced us that this last product is the result of the polymerisation under the influence of heat during distillation of butylene di-bromide, and is not present, to any considerable extent at least, in the original derivatives. Although the di-brom derivatives from ethylene dibromide ($C_2H_4Br_2$) to hexylene dibromide ($C_6H_{12}Br_2$) have been detected qualitatively, the separation of the various derivatives by fractional distillation *in vacuo* is not sufficiently sharp to give accurate quantitative results in regard to the amount of each constituent present. We therefore have, for the present at least, contented ourselves with calculating from the percentage of bromine in the di-brom derivatives the average number of carbon atoms in the molecule.

The results of the examination of a few samples of steel by the above method are shown in the following table.

The number of carbon atoms in the carbon molecule of the derivatives from the pure carbide, given in the following table, was obtained from the analysis of the gas by dividing the volume of carbon dioxide, produced from the explosion of olefines, by the volume of the olefines exploded.

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Name.	Heat Treatment.	Per Cent of Carbon of Steel.	Per Cent of Carbon as Derivatives.	Per Cent of Carbon as Gaseous Paraffins.	Per Cent of Carbon Unaccounted for.	Per Cent of Bromine in Derivatives.	Calculated Carbon Atoms in Carbon Molecule of Derivatives.
F	Annealed.....	0.55	37.1	33.6	29.3	72.56	4.32
F	Hardened and Tempered ..	0.55	25.0	. . .	. . .	75.65	3.67
C	Annealed.....	1.14	43.4	37.9	18.7	73.85	4.05
C	Hardened	1.14	29.0	48.6	22.4	77.61	3.31
D	Annealed.....	1.28	31.0	44.3	24.7	77.80	3.26
	Pure Carbide from D annealed	6.64	35.3	25.2	39.5	. . .	4.41

We know from a study of the above table that —

First, the average number of carbon atoms in the molecule derived from annealed steel decreases as the percentage of carbon increases.

Second, that in annealed steel the proportion of carbon escaping as gaseous paraffins, probably as methane (CH_4) or ethane (C_2H_6), increases as the percentage of carbon increases.

Third, that with the same steel, the effect of hardening seems to be to lower the number of carbon atoms in the molecule of derivatives, and to increase the proportion of carbon escaping as gaseous paraffins.

Fourth, from the percentage of carbon in the above steels it will be noted that "F annealed" must consist of pearlyte with ferrite, whereas "C annealed," and "D annealed," consist of pearlyte with increasing amount of cementite. The number of carbon atoms in the molecule of derivatives from "F annealed," and "pure carbide" isolated from the pearlyte in "D annealed," is nearly the same, 4.32 as compared with 4.41. Since the number of carbon atoms from "D annealed" is so much lower than from the pure carbide, the inference would be that cementite must yield derivatives of small numbers of carbon atoms in order to bring the average down to 3.26.

The results obtained from the examination of the steels, as shown in the preceding table, would indicate almost posi-

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tively that there are several carbides existing in steel, and that these carbides have different molecular weights. In all of the theories of the constitution of steel at present held, the formula CFe_3 is assigned to the carbide existing in annealed metal; this formula is merely an empirical one, representing only the results of ultimate analysis, without attempting to account in any way for molecular constitution; the same criticism would hold true of the formula CF_{24}, which represents the ultimate composition of martensite. Any theory which assigns so simple a formula as CFe_3 and CFe_{24} to the carbides of annealed and hardened steel, without taking into consideration the molecular weight of the carbides, simply because the carbides contain respectively 6.67 and 0.89 per cent of carbon, is as unsatisfactory as it would be to say that all hydro-carbons of the olefine series have the formula CH_2, because they all contain 85.71 per cent carbon.

In view of the fact indicated by our work on steel, I would venture to repeat again the hypothesis which I suggested in February 1897, before the American Institute of Mining Engineers. This hypothesis which I would offer, makes the fundamental assumption that carbon forms with iron a series of compounds which might properly be termed "ferro-carbons," on account of their similarity in structure to hydro-carbons. This series of ferro-carbons has the empirical formula $(CFe_3)_n$; or, as I prefer C_nFe_{3n}, this series of ferro-carbons are to be considered as being derived from the hydro-carbons of the olefine series with the general formula C_nH_{2n}, by the replacement of the H_2 by the bivalent group $\begin{matrix} \diagup \text{Fe}— \\ \text{Fe} \\ \diagdown \text{Fe}— \end{matrix}$. These ferro-carbons, dissolved in hydrochloric acid, yield as their primary products of solution the corresponding olefines and hydrogen. Thus, the first member of the ferro-carbon series, CFe_3, should yield as its products of solution methylene, CH_2, and hydrogen; the second member, C_2Fe_6, should yield ethylene, C_2H_4, and hydrogen; the third, C_3Fe_9, propylene, C_3H_6, and hydrogen, etc. As a matter of experience, however, we do not find the products of solution to be as simple as would be indicated by the above description, on ac-

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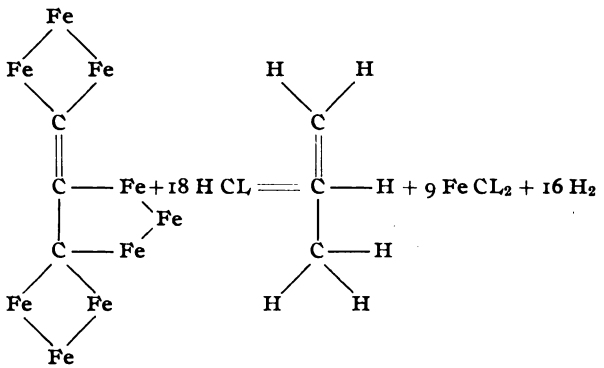
count of two well-known facts. First, because the olefines have a tendency to combine with nascent hydrogen to form the corresponding paraffin, according to the equation: $C_nH_{2n}+H_2=C_nH_{2n+2}$. This would readily account for so large a proportion of the carbon being found as methane (CH_4), with probably some ethane (C_2H_6), since methylene (CH_2) has as yet never been isolated, owing to its extreme chemical activity. The second factor which would tend to complicate the products of solution is the strong tendency of the higher olefines to polymerize under the action of acids; this tendency is quite marked in the case of propylene, and is very strong in the case of butylene and higher members.

The carbon unaccounted for in the above analyses remains as liquid hydro-carbon of high boiling-point, which does not pass over with the steam or hydrogen when the steel is dissolved. Whether these high-boiling hydro-carbons are primary products of solution, or result from polymerisation and hydrogenation of lower members of the series, we are not at present able to state.

The solution of the ferro-carbons would be shown in the following graphic equation:—



In annealed steel containing less than 0.89 per cent carbon, and consisting therefore of pearlyte with more or less ferrite, we would find the eutectic pearlyte consists of alternate laminæ of ferro-carbon, C_nFe_{3n}, and laminæ of iron, Fe_{21n}. This Fe_{21n} may be regarded as iron of crystallisation, the ferro-carbons occurring in pearlyte having a higher molecular weight than those occuring as cementite.

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When annealed steel is heated above Ar_1, the ferro-carbons C_nFe_{3n} dissolve in the iron of crystallisation, Fe_{21n}, forming what Professor Arnold has happily termed the "cryo-carbide" martensite, $C_nFe_{3n} \cdot Fe_{21n}$. Although this "cryo-carbide" has the same empirical formula as Professor Arnold's sub-carbide CFe_{24}, the formula $C_nFe_{3n} \cdot Fe_{21n}$ seems to be more in accord with our conceptions of chemical constitution and of the constitution of cryo-hydrates. The formula $C_nFe_{3n} \cdot Fe_{21n}$ accounts for the molecular weight of the existing ferro-carbons, and, although there is a definite amount (Fe_{21n}) of iron associated with the C_nFe_{3n}, just as there is a definite amount of water associated with a fixed amount of sodium chloride in the cryo-hydrate, all the 24n atoms of iron do not bear the same chemical relation to the *n* atoms of carbon, but 3n atoms of iron are combined with the carbon to form the ferro-carbon, the remaining 21n atoms of iron bearing the same relation to the ferro-carbon molecules that the water in the cryo-hydrate bears to the molecule of sodium chloride. When the ferro-carbons of high molecular weight dissolve in their iron of crystallisation, they probably begin to split up to a greater or less extent, yielding lower members of the series, this dissociation increasing with rise of temperature. The exact extent of this dissociation, under the influence of varying temperature, is still being investigated in this laboratory.

The points Ar_2 and Ar_3 may both indicate points of increased dissociation of the ferro-carbons, or one of these may denote an allotropic change in the iron. The dissociation of the ferro-carbons goes on with increased temperature, as is indicated by calculations of Baron H. von Jüptner,* who shows, from the depression of the melting-point of steel, that at the melting-point the carbon molecule does not contain more than two atoms of carbon.

When low-carbon steel is slowly cooled from very high temperatures, the dissolved ferro-carbons begin to polymerize and segregate with the iron constituting the cryo-carbide, until at Ar_2 the pure cryo-carbide martensite has separated completely from the surrounding ferrite. As the temperature falls to Ar_1, the ferro-carbons crystallize out with their iron of crystallization, constituting the eutectic pearlyte.

* *Journal of the Iron and Steel Institute*, 1898, No. II, page 247.

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Another argument in favor of the probable existence of a series of ferro-carbons is found in the behavior of steel toward nitric acid, in the well-known colorimetric method for the determination of carbon. It is well recognized among steel analysts that, in order to obtain reliable results, it is necessary that the unknown sample and the standard with which it is compared should not only have approximately the same carbon content, but should have been subjected to the same heat treatment. If there were but one carbide of iron, it is hard to understand why the depth of color obtained by dissolving in nitric acid should not be directly proportional to the amount of carbon in every case, so that low-carbon steels could be accurately compared with high-carbon standards. If, however, we assume the existence of several ferro-carbons, we can very readily see why it is necessary, in order that the nitro derivatives should be the same, that the standard and the unknown should have approximately the same percentage of carbon and the same heat treatment, since large variations in the percentage of carbon and varying heat treatments alter the members of the ferro-carbon series in existing steels.

Again, because the ultimate composition of the carbide in pearlyte is probably the same as that constituting cementite, the statement is usually made that pearlyte consists of alternate laminæ of cementite and ferrite. It is hard to conceive how this can be the case if there were but one carbide of iron in annealed steel, and this had the formula CFe_3. We might, if this were true, justly expect that this carbide and the crystallized iron associated with it should have the same solubility as the two constituents when separate. Cementite remains bright under the action of iodine and of nitric acid, as does also ferrite, yet pearlite, which is said to consist of alternate laminæ of these two constituents, neither of which is acted upon by the reagents in question, is readily colored by both. If, however, we conceive that the carbides in pearlyte and in cementite are not the same, but different members of a ferro-carbon series, we can then readily understand the difference in solubility of the two substances.

Hydrogen must be regarded as very closely related to the metals, and it is hard to understand how there is any inherent reason why the formation of complex molecules of carbon

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should be confined to compounds of carbon with hydrogen and the non-metallic elements like oxygen, nitrogen, etc.; why hydrogen in various hydro-carbons should not be capable of being replaced completely by metals forming what might properly be called metallo-carbons. If we succeed in establishing the probable existence of ferro-carbons, it is proposed to take up the study of some of the other carbides, in order to determine whether or not other series exist.

THE PRESENT POSITION OF THE SOLUTION THEORY OF CARBURIZED IRON.*

By A. STANSFIELD.

Introduction.

IN view of the large amount of attention that has recently been devoted to the constitution of steel and other forms of carburized iron, and of the importance of the solution theory in explaining the mutual relations of carbon and iron, it appeared desirable that an attempt should be made to embody in an accessible form the main features of this theory. It is usually admitted that metallic alloys behave as saline solutions do, and bringing carburized iron into line with alloys is rendering a good service to industrial and theoretical chemistry. I need not remind members of this Institute that a solution need not be a liquid, but that in certain solids, especially when they are at a high temperature, an added element tends to diffuse itself uniformly through the mass, from which, however, as from the liquid solution, it may be crystallized out on cooling.

Such a solution is white-hot steel. It has, in fact, been abundantly proved that a solution need not necessarily be fluid. A mass may be solid and yet retain many of the characteristics of a solution. It has, for instance, been shown† that molecular changes have precisely the same effect in producing crystallization from a solid solution that

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* Iron and Steel Institute, August 1899 meeting.

† Victor Rothmund, *Zeitschr. f. Physikal. Chem.*, 1897.

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the freezing of an ordinary solution has. Just as a body dissolved in water is separated by solidification or freezing, so a body dissolved in a solid may "fall out" when a molecular change takes place. The molecular changes of iron are, therefore, of the first importance in relation to the solution theory of solidified carburized iron.

The solution theory of carburized iron affirms that this substance is, when fluid, a solution of carbon in iron, and that under certain conditions the solidified mass also forms a solid solution. It further affirms that these liquid and solid solutions obey the ordinary laws of solution, which have been fully studied in the case of aqueous, saline, and organic solutions. The solution theory can, therefore, be invoked to explain both the mode of solidification of carburized iron and the molecular changes that take place after solidification. The experimental data which enable the theory to be applied are mainly afforded by the pyrometer and the microscope used in conjunction with chemical analysis, but all the other chemical and physical properties of carburized iron must be studied for the sake of the contributory evidence they afford.

The solution theory of carburized iron can be most readily understood by reference to the "Freezing-point Curves" contained in Sir William Roberts-Austen's Fifth Report to the Alloys Research Committee of the Institution of Mechanical Engineers. A diagram of these curves has been lent by that Institution to illustrate this paper. (See next page.)

The diagram embodies the information gained from a large number of cooling curves of carburized iron, which have been obtained by means of the autographic recording pyrometer. It indicates, for each percentage of carbon, the temperatures at which evolutions of heat would occur in a slowly cooling mass.

The Solidification of Carburized Iron.

The freezing temperature of pure iron has not been determined exactly, but we may for the present consider it to be 1600° C.

Carbon will dissolve in molten iron just in the same way as common salt dissolves in water, and in both cases the

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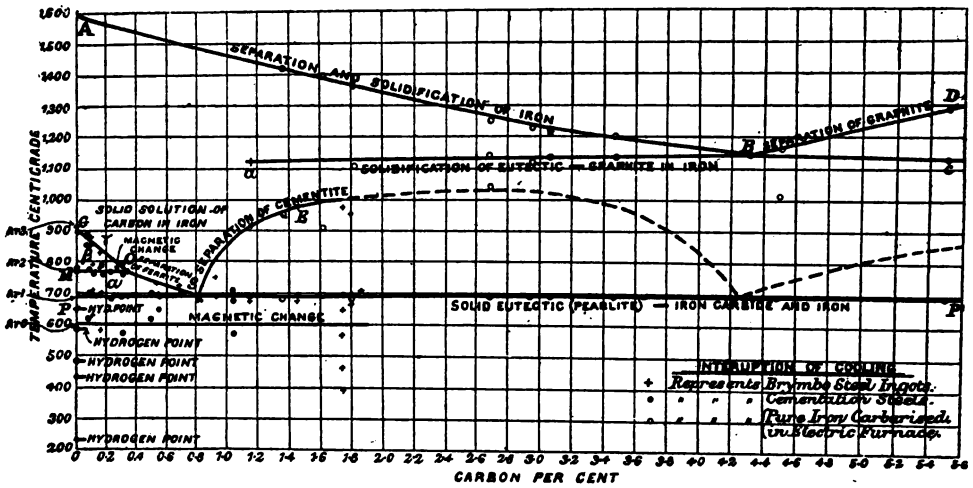
freezing-point of the solution is lower than that of the pure solvent.

The freezing temperature is continuously lowered with further additions of carbon, as is indicated by the line AB, until at about 4.3 per cent* the maximum fusibility is attained, and further additions of carbon cause a rise (BD) in the freezing temperature. This is exactly analogous to the behavior of a salt solution.

The first solid to separate from a freezing salt solution is pure ice if the solution is weak, and pure salt if the solution



is strong; similarly pure carbon, in the form of graphite, separates as "kish" from a strong solution of carbon in iron. The first solid to separate from a dilute solution of carbon in iron is either pure iron, or a solid solution of carbon in iron which contains, however, less carbon than the liquid solution from which it solidified.

The residual mother-liquor from which either the graphite or the iron is separating becomes correspondingly poorer or richer in carbon until its composition approximates to 4.3 per cent of carbon. If the solution contains a little more carbon

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* Percy found that 4.6 per cent of carbon was the maximum that pure iron could contain without separation of kish.

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than 4.3 per cent, graphite will separate out, reducing the composition of the residue to below 4.3 per cent. After this some iron will separate out, thus raising the percentage of carbon in the residual liquid, so that, on the whole, the composition remains constant. Thus it will be evident that iron containing 4.3 per cent of carbon, whether originally prepared of this composition, or resulting naturally as a residual liquid or eutectic during the cooling of a carbon iron solution, must solidify as a mixture of iron and graphite. It will also be evident that the temperature at which it solidifies must be a constant one.

The horizontal line *aBc* shows that in iron containing more than about 1.2 per cent of carbon there is a residual liquid which solidifies at a constant temperature of about 1130° C. The microscope usually shows the presence of graphite in any alloy in which a point in the cooling curve has been revealed at 1130° C. The very small quantities of graphite indicated in steels having less than about 2 per cent of carbon may sometimes be converted into carbide during the subsequent cooling from this temperature. The iron, on the other hand, is always subject to changes during the cooling, and these will be considered subsequently.

It has been abundantly proved that eutectic alloys and the so-called cryohydrates (their analogues in saline solutions) are not chemical compounds, as was supposed at one time, but are true solutions when liquid, and are mechanical mixtures of two constituents when solid.

The constitution of the solidified iron must now be considered. Does the iron separate in a pure state, or does it hold some carbon in solid solution? The fact that the eutectic line ABC has not been traced in steels containing less than about 1.2 per cent of carbon, shows that at 1130° C. iron can hold 1.2 per cent of carbon in solid solution. It might naturally be inferred from this that at the higher temperatures at which the iron originally solidified it would contain *more* carbon in solid solution. There are, however, certain considerations which do not favor this view. One of these considerations arises from the fact that at rather lower temperatures, such as 1000° C., iron can hold *more* than 1.2 per cent of carbon in solid solution, so that there is clearly some in-

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fluence at work which causes, as the temperature rises, a *decrease* in the amount of carbon dissolved by the iron.

Now at 1130° an excess of carbon in iron would tend to separate as graphite, but at 1000° it would separate as the carbide of iron, Fe_3C, and it has been suggested* that the carbon is dissolved in the iron as carbide at lower temperatures, and as free carbon at higher temperatures. There is, fortunately, additional evidence on this question, and it tends in the same direction.

An equation devised by Van't Hoff renders it possible to calculate from the slope of the line AB the number of atoms in each molecule of carbon dissolved in molten iron.

Baron von Jüptner† has made this calculation, and finds that the molecule of dissolved carbon contains two atoms in the case of fluid steel, and that in fluid pig iron there are an equal number of two and three atom molecules. The formula of Van't Hoff is, however, only correct for very dilute solutions, and should not be used in the case of pig iron. Heycock und Neville‡ give a modification of this formula, which is more suitable for such solutions. The author has applied Heycock and Neville's formula, and finds that the molecule of dissolved carbon contains two atoms both in fluid steel and fluid pig iron.§

All these calculations are dependent on the assumption that the iron separates in a pure state on solidification. If the iron contained dissolved carbon, it would involve the existence of a molecule containing less than two atoms.

As, however, the molecule could never contain less than *one* atom, a limit is put to the amount of carbon which the iron can possibly hold on solidification. If the molecule were monatomic, the iron would contain on solidification approximately one-half the amount which it contained when fluid.

Thus an examination of the lowering of the freezing-point of iron, produced by the addition of carbon, shows that the

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* Baron von Jüptner, *Journal of the Iron and Steel Institute*, No. I, 1898, page 235.

† *Journal of the Iron and Steel Institute*, 1898 and 1899.

‡ *Journal of the Chemical Society.*

§ These calculations involve a knowledge of the latent heat of fusion of iron, which has been taken as 20 calories.

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iron contains after solidification an amount of carbon which is certainly not more than one-half of the amount in the solution from which it separated.

An examination of the amount of heat evolved at 1130° C., by cooling masses containing varying percentages of carbon, strongly supports the view that the iron retains on solidification about one-half the percentage of carbon of the liquid from which it solidified.*

For the present, then, the author wishes to advance the hypothesis that the carbon in fluid iron is monatomic, and that the iron which separates on cooling contains about one-half the percentage of carbon of the liquid from which it crystallized. It will follow on this hypothesis that the eutectic consists of a mixture of graphite and of iron containing about 2.2 per cent of carbon.

Formation of Carbide of Iron.

It has already been mentioned that the carbon which remains in solid solution in the iron probably enters into combination with it when it is cooled below about 1000°, forming the carbide Fe_3C. The evidence upon which this rests is as follows.

1. The pure carbide of iron, Fe_3C, when heated to about 1050° C. or 1100° C., is dissociated, forming graphite and iron containing some dissolved carbon. It might, however, be expected that Fe_3C in solution would be able to exist at still higher temperatures than the free carbide, on account of the osmotic pressure of the released carbon and iron; the extent to which this takes place requires investigation.

2. The evidence already mentioned, that the solubility of carbon in solid iron appears to diminish, or at any rate not to increase so rapidly, as the temperature is raised above about 1050° C.

3. Evidence on this point may also be gained by a consideration of the nature of the constituent austenite, which Mr. Osmond has added to our already long list. This may be

* The only alternative to this is to suppose that a large amount of combination takes place above 1130° C. between the solidified iron and the still fluid carbon.

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obtained by heating a high carbon steel to a temperature above 1050° C. and quenching rapidly in iced brine. The steel is then found to possess a peculiar zigzag structure (when examined under the microscope), and consists of two elements — a hard material resembling martensite, and a softer material, "austenite," which appears to be non-magnetic.

Mr. Osmond has recently shown* that austenite resembles manganese or nickel steels, and, like them, may be rendered magnetic by immersion in liquid air for some minutes. The density of the steel containing austenite is about as great as that of annealed steel, but upon cooling in liquid air it becomes less. The hardness increases at the same time, and approximates to that of martensite.

Mr. Osmond explains that when γ-iron contains a large amount of (dissolved) carbon (as is the case in high carbon steels above 1050° C.), very rapid quenching enables (part of) the iron to remain in the γ form. The subsequent cooling in liquid air enables the γ-iron to change to the usual (α?) form it assumes in quenched steel. Baron von Jüptner† advances the explanation that when high carbon steel is heated to 1050° C., the cementite partly dissociates, producing a solution of free carbon in iron.

The explanation of Mr. Osmond satisfactorily accounts for the non-magnetic properties of austenite, but that of Baron von Jüptner may also be invoked to explain its want of hardness.

Formation of Graphite.

The formation of graphite from the eutectic of the molten carburized iron, and also at temperatures indicated by the line BD in irons particularly rich in carbon, has been already explained. The mode of formation of the variety of graphite known as "temper-carbon," and the reason for the comparative absence of cementite in rich carburized iron, has yet to be considered. At temperatures between about 1000° C. and 1130° C., the dissolved cementite tends to dissociate, and the carbon may be liberated as

* *Comptes Rendus*, 1899. *The Metallographist*, Vol. II, page 261.
† *Journal of the Iron and Steel Institute*, 1898.

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"temper-carbon" either directly or after an intermediary stage of simple solution in iron. Conversely, at temperatures below 1000° C., the graphite gradually dissolves in the iron and is converted into cementite, which may fall out of solution on further cooling.

In high carbon steel the *mechanical pressure* engendered by the separation of graphite (which occupies more space than when in solution) probably limits the formation of graphite during the limited range of temperature in which it can be liberated. In the case of highly carburized pig iron, the large number of flakes of graphite formed on solidification constitute nuclei around which more graphite can separate, and, as the mass is more open, internal pressure cannot well be set up. It is, therefore, difficult to determine the degree of solubility of graphite in solid iron, as it varies so much with the conditions under which the separation takes place.

The Allotropic Changes of Iron and their Influence on the Solid Solutions of Iron and Cementite.

Solid iron differs from most other metals, as it can exist in more than one molecular form. The change from liquid iron to solid iron has already been considered, but there remain the changes, almost as profound, from γ-iron to β-iron, and from that to α-iron.

In pure iron these changes take place on cooling; from γ to β at about 895° C. (the Ar_3 point), and from β to α at about 765° C. (the Ar_2 point).* The presence of dissolved cementite lowers the temperature at which these changes occur in precisely the same manner as the presence of dissolved carbon in molten iron lowers the temperature of its freezing-point.†

To take first steel containing less than 0.34 per cent of

* The allotropic varieties of iron are now well known, but it may be mentioned that γ-iron is non-magnetic, and dissolves carbon and cementite; β-iron is non-magnetic, but does not dissolve cementite; while α-iron is magnetic, and does not dissolve cementite.

† Mr. Osmond makes a similar statement at the end of the paper already referred to, comparing the lowering of these allotropic changes to the lowering of the freezing-point of a solvent.

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carbon, in which both Ar_2 and Ar_3 occur; separation of β-iron commences at a temperature denoted (for each percentage of carbon) by the line GO. Iron in the γ form will dissolve about 1 per cent of carbon (as cementite) at 850°, but β-iron will dissolve scarcely any, so that the separated β-iron, being practically free from carbon, undergoes the change to α-iron at the normal temperature of 765° C. Meanwhile, as the iron falls out, the residual solution of cementite in γ-iron becomes richer in cementite, until at 690° C. it is saturated, forming a eutectic solid solution, and the cementite and the iron (in the α form) separate out side by side to form the well-known pearlyte. The evolution of heat at 690° C. which marks this change is known as the Ar_1 point. The molecular mechanism producing the lowering of the allotropic change point, Ar_3, is analogous to that producing the lowering of the freezing-point of iron, and the formulæ of Van't Hoff and of Heycock and Neville are equally applicable to both cases. In calculating the molecular weight of the dissolved carbide from the lowering of the Ar_3 point, the temperature and latent heat of fusion of pure iron must be replaced by the temperature and by the heat rendered latent when pure electro-iron changes from β to γ-iron at Ar_3.

Sir W. Roberts-Austen, in his Presidential Address,* stated, as the result of such a calculation, that the molecule of dissolved carbon consisted of three carbon atoms, which would mean, supposing that the carbon exists in solution as cementite, that it would have the formula $3(Fe_3C)$; and although a later calculation, in which certain corrections were introduced, results in the smaller figure of two carbon atoms in the molecule, $2(Fe_3C)$, the data involved in the calculation are not yet sufficiently exactly determined to enable a very definite statement to be made.†

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* *Journal of the Iron and Steel Institute*, No. I. *The Metallographist*, Vol. II, page 340.

† The amounts of heat rendered latent during the changes of iron from α to β and from β to γ were obtained from cooling curves of electro-iron (like the one published in the fifth report of Sir W. Roberts-Austen to the Alloys Research Committee of the Institution of Mechanical Engineers). The apparent rise of temperature of the iron was measured, and, after making allowance for the probable loss of heat, it was multiplied by the specific heat of the iron. The Ar_3 change from γ to β

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If the steel contains 0.34 per cent of carbon, the Ar_3 point occurs at the same temperature as the Ar_2 point, and further additions of carbon result in a lowering, to temperatures indicated by the line OS, of what may be described as the combined point $Ar_{2.3}$. In such steels the excess of iron separates directly in the α form, and the residual solid solution is decomposed as before at the point Ar_1 at 690° C. The theory connecting the lowering of the point $Ar_{2.3}$ with the number of molecules of Fe_3C in unit volume of the solution, is less simple than when the point Ar_3 was alone concerned. A calculation may, however, be made on the assumption that the osmotic pressure required to produce the observed lowering is equal to the sum of the pressures which would be required to lower each point, Ar_2 and Ar_3, separately.

The number (2.7) obtained in this way for the number of carbon atoms in the molecule does not agree at all with the number (2.0) already mentioned. This discrepancy is represented graphically on the diagram by the decided change in direction from the line GO to the line OS, a change which seems too great to be fully accounted for by the influence exerted by the comparatively small change Ar_2 upon the direction of the line OS. The evolution of heat at Ar_2 is, however, less abrupt, and therefore less easy to measure, than that at Ar_1. The specific heat of iron, moreover, (which is involved in these measurements) probably varies extremely with the temperature, and more exact measurements of these evolutions of heat, which represent the latent heat of the allotropic changes, may be expected to produce a better agreement between the results.

When the amount of carbon in the steel has reached an amount which is between 0.8 per cent and 0.9 per cent, the composition is the same as that of the solid eutectic. Under these conditions the solid solution of cementite and of γ-iron cools down as a whole, until, at the temperature of 690° C., the cementite and ferrite are mutually saturated, and split up into the characteristic pearlitic structure of cementite and α-iron.

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caused an apparent rise of temperature of 14.3° C. (including the correction), and this represents 2.86 calories. The Ar_2 change from α to β corresponded to 4.2° C. or 1.0 calories; but the latter figure may possibly be quite inaccurate.

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Still further additions of carbon cause the steel on cooling to become saturated with cementite, which begins to separate at temperatures indicated by the line SE. The residual solid solution, therefore, becomes poorer in cementite, until at 690° C. it has the eutectic composition.

The conclusions which the author has arrived at with respect to the atomic complexity of carbon in iron are these: —

The carbon in molten iron is in a state of simple solution; the molecule of carbon must then contain one or two atoms, and is probably monatomic. The solidified iron is in the γ state and contains free carbon in solution. The molecular weight of this carbon has not been discussed, but it is probably the same as that in the molten iron.

The carbon in solid solution combines with iron, on cooling, to form a carbide, which is probably expressed by the formula $2(Fe_3C)$.

When, on further cooling, this carbide falls out of solution as cementite, its formula may become more complicated; the solution theory affords no information on this point; but Sir W. Roberts-Austen stated in his Presidential Address that the nature of the products of its solution in acids led to the conclusion that the molecule may contain six atoms of carbon, and is at least as complex as would be indicated by the formula $6(Fe_3C)$.

There appears to be a belief that the solution theory is in a sense opposed to, and has gone far to, supplant the older allotropic theory, but this paper will, it is hoped, effectually dissipate such an error, as it shows how entirely the solution theory of the relations of carbon and iron involves the allotropic changes with which the distinguished name of Osmond is so inseparably connected.

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STEELS FOR MAGNETS.*

By F. OSMOND.

IN order that a certain steel may give useful permanent magnets, it is necessary and sufficient that its critical points be located at a temperature below 350° C. or thereabout, and above the lowest temperature to which the metal will ever be subjected.

There are two ways to meet this requirement: 1st, by hardening carbon steels; 2d, by adding to the steel suitable proportions of some foreign element (Mn, Ni, Cr, W,) which directly, or indirect through their action upon the carbon, lower sufficiently, during slow cooling from a high temperature, the critical points of iron.

The steels which owe their magnetic properties to the hardening operation have been studied at length and with great skill by Mrs. Curie.†

The steels of the second group have not been so well studied. In Mrs. Curie they are represented by a single type containing 7.70 per cent of tungsten. This group, however, also includes steels containing from 10 to 25 per cent of nickel, from 3.5 to about 7.5 tungsten, from 5 to about 15 of chromium, in short, all steels which assume spontaneously properties similar to those of hardened steel, and which are for that reason called "self-hardening." Like hardened steel, self-hardening steels are capable of assuming two conditions: a hard and a soft one; they become hard when allowed to cool from a temperature higher than their critical points during heating, and they are softened by reheating to a temperature lower than that of the critical points.

The study of such metal calls therefore for a double inquiry: the treatment which confers to the steel the best magnetic properties must be ascertained as well as the treatment which will make it possible to perform the necessary machine work. The softening or tempering process is well known, and it is more especially the treatment required to convert

* *Comptes Rendus*, June 19, 1899. Inquiry carried on in the Sorbonne Laboratory of General Chemistry.

† *The Metallographist*, Vol. I (1898), pages 107, 229 and 274.

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the steel into a permanent magnet of good quality which calls for further investigation.

The samples which were examined, and which come from the collection of Mr. Hadfield, had the following compositions and dimensions:

	C.	Mn.	Ni.	Weight gr.	Length mm.
1.	0.23	0.93	15.48	10.670	36.7
2.	0.19	0.93	19.64	11.285	38.5
3.	0.16	1.00	24.51	12.345	37.6
4.	0.45	4.00		11.135	39.2
5.	0.32	5.67		11.754	39.2
6.	0.46	7.80		11.616	39.6

Each sample was brought into its hardened condition by reheating it above its critical points, followed by slow cooling. It was then tempered at gradually increasing temperatures. After each treatment the steel bar was tested with a file, and magnetized by means of an electro-magnet traversed by a current of 5.5 amp. It was then placed in a constant position before a reflection magnetometer.

The results of these magnetic tests are tabulated below.

The columns marked *t* indicate, in the order followed, the successive temperatures to which the bars were heated; the columns marked *d* and *d'* show the deviations read on the scale placed at a distance of one meter from the galvanometer, respectively immediately after the magnetization and after a rest of fifteen minutes.

NICKEL STEELS.

15.48 per cent Ni.			19.64 per cent Ni.			24.51 per cent Ni.		
t.	*d.*	*d'.*	*t.*	*d.*	*d'.*	*t.*	*d.*	*d'.*
837	132.0	118.5	837	121.0	106.0	842	112.0	110.7
770	141.0	126.5	770	126.6	114.5	723	124.6	123.0
710	146.0	127.9	710	131.5	118.4	158	115.1	114.8
634	155.9		634	169.0		295	92.8	92.1
105	154.8	152.0	105	168.0	163.7	399	62.9	62.9
214	139.3	138.9	214	150.9	150.0	454	61.8	61.2
303	123.7	123.3	303	132.3	132.5	513	79.9	79.4
370	98.1	98.0	370	104.9	104.9	585	152.7	151.1
415	78.0	78.0	415	82.9	82.9	648	152.0	151.0
475	64.0	64.0	475	72.1	72.1			
514	64.8	64.5	514	78.4	78.0			
565	149.0	136.0	565	181.0	171.0			
603	164.0	149.8	603	175.0	165.5			

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MANGANESE STEELS.

4.00 per cent Mn.			5.67 per cent Mn.			7.80 per cent Mn.		
t.	*d.*	*d'.*	*t.*	*d.*	*d'.*	*t.*	*d.*	*d'.*
755	161.8	142.2	755	148.7	148.1	750	1.2	
−190	182.9	166.1	125	138.0	136.0	−190	151.9	144.0
125	164.3	162.5	355	135.0	123.2	157	163.2	163.2
355	70.4	69.1	574	165.0	153.5	315	177.0	176.4
594	95.5	93.5				479	182.9	182.0
						618	217.5	214.8
						−190	234.9	230.4
						594	205.9	205.9
						−190	210.0	209.4
						534	204.8	204.0

As a standard of comparison, a similar bar of the ordinary quality employed for the manufacture of magnets was used. It contained 0.71 per cent of carbon, 0.73 of manganese, 3.47 of tungsten; it had a length of 39.5 mm., and weighed 11.612 grams. Quenched in water from a cherry red temperature, it gave a deviation of 197.1 mm., which was reduced to 191 mm. after fifteen minutes.

Among the steels studied, several exhibit nearly as good magnetic properties as the standard's, and one of them superior ones, at least under the conditions prevailing during the testing.

The nickel steels and the manganese steel containing 4 per cent of that element exhibit a minimum residual magnetism which nearly coincides with the minimum hardness.

The proper treatment of these steels is, therefore, as follows: 1st, tempering after forging to a temperature a little below that of the critical points; 2d, the necessary machine work; 3d, reheating to just above the critical points, in order to secure the maximum of magnetic intensity; 4th, tempering to about 100° C., to render the magnetization more stable.

In the sample containing 5.67 per cent of manganese the minimum of magnetic intensity is feebly marked; in its soft condition this sample still gives fairly good magnets.

The sample containing 7.80 per cent of manganese twice cooled in liquid air and tempered at a dark red after each cooling, yields remarkable magnets, but it cannot be appreciably softened.

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The interest which attaches to these self-hardening steels is due to the fact that the hardening operation is avoided and that the magnetic properties are uniform throughout the mass. The subject is worthy the attention of physicists.

ON THE CHANGES OF STATE IN IRON AND STEEL.*

By H. LE CHATELIER.

THE discovery by Gorre and Barrett of the recalescence of steel, and the discovery by Osmond of the two transformations of iron have been the starting-point of numerous researches. Much obscurity, however, still remains; the systematic use of very rapid heatings and coolings has resulted in exagerating the importance of the retardations during the transformations. It is difficult, under such conditions, to distinguish what belongs to the reversible phenomenon itself and what to the passive antagonistic resistances.

My intention was, in the present inquiry, to study these transformations by measuring the dilatations at temperatures, if not absolutely stationary, at least varying very slowly.

1. *The Point of Recalescence of Steel.* — This transformation, the best known of the three, corresponds to a point similar in every respect to the lowest freezing-point of mixtures of ice and salt; during this retardation a eutectic mixture is formed, at the expense of a solid solution (martensite) of cementite and ferrite, i.e. of the carbide Fe_3C and of carbonless iron, the eutectic mixture being made up of thin plates alternately of cementite and ferrite. It was natural to suppose that during this critical point a single change of volume took place, as is the case during the fusion or dissolution of any substance, and that on either side of this point the dilatation would be found to follow a uniform law. Such, however, is not the case. Two changes of dimensions occur successively, in opposite direction, and which nearly compensate each other when the steel contains about 0.90 per cent of carbon.

* *Comptes Rendus*, July 31, 1899.

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The following table shows the dilatations of such a steel while its temperature is increasing. The dilatations are expressed in millimeters per 100 millimeters of length. The line marked *difference* expresses the difference between the dilatation recorded and what it would have been if it had continued to vary at its normal rate.

FIRST EXPERIMENT.

Temperature . .	600°	760°	775°	800°	830°	860°	950°
Dilatation . . .	0.82	1.05	0.96	1.00	1.12	1.20	1.34
Difference . . .	"	"	−0.11	−0.11	−0.06	"	"

SECOND EXPERIMENT.

Temperature . .	315°	500°	690°	775°	800°	850°	900°
Dilatation . . .	0.35	0.63	0.93	1.06	0.98	1.14	1.25
Difference . . .	"	"	"	"	−0.12	−0.04	−0.01

The total maximum change reaches 0.115 mm., which is twice that found by Mr. Svedelius.* In the experiments of Mr. Svedelius, however, the very rapid heating lasted only two minutes, while in mine the heating lasted several hours. The coincidence of the dilatation curve above the point of transformation with the extension of the lower curve results from the immediate succession of two changes, equal and of opposite direction. Such a fact is at present without any known analogy.

2. *Magnetic Transformation of Iron.* — This transformation, according to the investigations of Messrs. Curie and Osmond on the magnetism of iron, is not sudden, as is the case in all similar phenomena. For two reasons, however, the experimental results are not conclusive: it has not been possible to separate the reversible phenomenon of the retardations during the transformation, and, moreover, magnetism must, like heat and mechanical work, be one of the determinative conditions of the transformations, so that the continuity or discontinuity of the transformation will depend upon the conditions prevailing during the experiment. It is why a liquid has a definite boiling point under constant pressure, while it has none under a constant volume.

My efforts to ascertain the changes of dimensions corresponding to this transformation have failed; it must therefore

* *Phil. Mag.*, Vol. XCVI (1898).

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be very slight, less than 0.01 mm. in a length of 100 mm., which is the limit of precision of my observations.

Similar researches carried on with nickel have shown that the transformation corresponding to the loss of the magnetic properties certainly occurs continuously, covering an appreciable range of temperature, between 350° and 380° C.

Proceeding by analogy we are led to infer, therefore, that with steel, as with nickel, the transformation takes place progressively, even outside of all magnetic fields. This is a very important fact, for it constitutes a unique instance among all the known transformations of definite solid bodies. Liquid and vapors only exhibit similar phenomena; the transformation of melted sulphur for instance, and that of the vapors of peroxide of nitrogen. A dimorphous transformation in the crystalline state is always discontinued; continuity in the transformations appears to be characteristic of an amorphous condition.

3. *Upper Transformation of Iron.* — This transformation, which is sharply marked in the case of electrolytic iron, exhibits some inexplicable anomalies which have sometimes led to the supposition that it was due to the presence of hydrogen or sulphur. I have also in my experiments detected such anomalies. The temperature or the transformation and the corresponding changes of dimensions vary in a way which cannot at present be defined. The following figures were obtained with a sample of iron containing 0.05 per cent of carbon.

Nature of the atmosphere.	Temperature of the transformation.	Change of length.
	First Experiment.	
	mm.	
Air	0.25	from 840 to 930
Pure hydrogen	0.26	from 900 to 970
	Second Experiment.	
	mm.	
Ordinary hydrogen	0.26	from 840 to 860
" "	0.25	from 900 to 1000
Air	0.20	from 950 to 1025
Ordinary hydrogen	0.14	from 925 to 975

The variability of this transformation renders this phe-

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nomenon at least as abnormal as the two first transformations.

The conclusion from these researches is that, although the application of the laws of polymorphism and of dissolution to the study of the properties of iron has often proved a precious guide, it must be remembered that the metal possesses remarkable peculiarities, which renders impossible a complete identification of its properties with those of other bodies. Long researches will still have to be carried on before it will be possible to reach definite conclusions.

It is not impossible that the explanation of these peculiarities will be found in the existence of a double fusibility, similar to that which has been discovered in selenium by Lehmann and Tammam. In its crystalline state this element is stable only between 60° and 214°C. Outside of these limits it is only the amorphous variety (vitreous and liquid) which is stable.

COLORS OF HEATED STEEL CORRESPONDING TO DIFFERENT DEGREES OF TEMPERATURES.*

By MAUNSEL WHITE AND F. W. TAYLOR.

THERE is, perhaps, nothing more indefinite in the industrial treatment of steel, than the so-called color temperatures, and as they are daily used by thousands of steel workers, it would seem that a few notes on the subject would prove of general interest.

The temperatures corresponding to the colors commonly used to express different heats, as published in various text books, hand books, etc., are so widely different as given by different authorities, it is impossible to draw any definite or reliable conclusion. The main trouble seems to have been in the defective apparatus used for determining the higher temperatures. The introduction of the Le Chatelier pyrometer within the last few years has placed in the hands of the scientific investigator an instrument of extreme delicacy and

* Read at the December 1899 meeting of the American Society of Mechanical Engineers.

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accuracy, which has enabled him to determine the temperatures through the whole practical range of influence, and led to the establishment of new melting and freezing-points of various metals and salts, which are now accepted as the standard in all scientific investigation. There have not, however, been published any results with the Le Chatelier pyrometer seeking to establish a correspondence of temperatures with color heats.

The first work done in this line, of which we are aware, is that of Prof. H. M. Howe, some eight or nine years ago. His results, however, have not been published, and with his kind permission we are enabled to give them here:

Dull red	625° to 550° C.	1022° to 1157° Fahr.
Full cherry	700°	1292°
Light red	850°	1562°
Full yellow	950° to 1.000°	1742° to 1832°
Light yellow	1050°	1922°
Very light yellow	1100°	2012°
White	1150°	2102°

The nomenclature used for color heats differs with different operators, but in our investigation we have adopted that which seems more nearly to represent the actual color corresponding to the heat sought to be represented. We have found that different observers have quite a different eye for color, which leads to quite a range of temperatures covering the same color. Further, we have found that the quality or intensity of light in which color heats are observed — that is, a bright sunny day, or cloudy day, or the time of day, such as morning, afternoon, or evening, with their varying light — influence to a greater or less degree the determination of temperatures by eye.

After many tests with the Le Chatelier pyrometer, and different skilled observers working in all kinds of intensity of light, we have adopted the following nomenclature of color scale with the corresponding determined values in degrees Fahr. as best suited to the ordinary conditions met with in the majority of smith shops:

Dark blood red, black red	990°
Dark red, blood red, low red	1050°
Dark cherry red	1175°

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Medium cherry red	1250°
Cherry, full red	1375°
Light cherry, bright cherry, scaling heat,* light red	1550°
Salmon, orange, free scaling heat	1650°
Light salmon, light orange	1725°
Yellow	1825°
Light yellow	1975°
White	2200°

With the advancing knowledge of, and interest in, the heat treatment of steel, the foregoing notes, it is hoped, may prove of some value to those engaged in the handling of steel at various temperatures, and lead to further and wider discussion of the subject, with a view to the better understanding and more accurate knowledge of the correct temperatures. The importance of knowing with close approximation the temperatures used in the treatment of steel cannot be overestimated, as it holds out the surest promise of success in obtaining desired results.

This demand for more accurate temperatures must eventually lead to the use of accurate pyrometric instruments; but at present the only available instruments do not lend themselves readily to ordinary uses, and the eye of the operator must be largely depended upon; therefore, the training of the eye, by observing accurately determined temperatures, will prove of much material assistance in the regulation of temperatures which cannot be otherwise controlled.

THE COLOR-NAMES FOR HIGH TEMPERATURES.†

By HENRY M. HOWE.

AN important paper by Messrs. Maunsel White and F. W. Taylor‡ brings up the question of temperature, in degrees Fahrenheit or Centigrade, which corresponds to each of the color-names "A dull red," "A light yellow," etc., etc.

* Heat at which scale forms and adheres, i.e. does not fall away from the piece when allowed to cool in air.

† *Engineering and Mining Journal*, January 20, 1900.

‡ This paper will be found in the present number of *The Metallographist.*

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They give the temperatures which in their own opinion, as the result of observing carefully the color impression produced on their own eyes and those of other experts by known temperatures as determinded by the Le Chatelier pyrometer, correspond to these various color-names.

In the accompanying table I give their results, together with Pouillet's and my own,* each line being so arranged as to contain that color-name of Pouillet and my own which correspond most closely to a given color-name of Messrs. White and Taylor.

While my own results agree in all cases within 55° C. (98° F.) with those of White and Taylor, Pouillet's differ from theirs by much larger amounts, ranging from 172° to 467° F. On an algebraic average, my results differ from theirs by some 28° C. (say 51° F.), while Pouillet's differ from theirs by some 167° C. (300° F.), or five times as much.

The difference between my results and those of White and Taylor can readily be referred to the personal equation. First, one and the same red-hot object will produce on different eyes different impressions of color; and secondly, different minds will select different names for one and the same impression of color. Hence it is not to be expected that the temperatures which we have picked out as typical of the different color-names should agree accurately. Next, each name really corresponds, not to a single precise degree Fahrenheit, but rather to a range of temperature. Now even if by a series of coincidences we had agreed exactly as to the limits of each range, we should hardly agree as to the individual degree Fahrenheit in any range which typifies most accurately the name of that range.

But the difference between Pouillet's results and those of White and Taylor are far too great to be referred to the causes just mentioned, and must, I think, be referred to gross errors of color-naming on Pouillet's part.

In view of the reasonable agreement between the results

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* My results were obtained in 1891 by means of the Le Chatelier pyrometer, but were not published till Messrs. White and Taylor published them for comparison with their own. Their results deserve much greater weight than mine, because based on the color-impressions of several different people.

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of White and Taylor and my own, they may be regarded as in a measure confirming each other and opposing those of Pouillet. The latter, while in my opinion widely at variance with the common acceptation of the color-names, are the ones usually given in text-books. I wish to remonstrate against their further retention, because I believe them to be far wrong and extremely misleading, and I recommend that the results of Messrs. White and Taylor be adopted in their stead.

Of course, when we attempt to displace data which have been so widely circulated, and replace them with others, we must proceed carefully, especially in view of the high standing of Pouillet, who certainly played a most important part in advancing pyrometry. Barus, in his late classical work on pyrometry, has justly testified to the worth of Pouillet's work.* And, from comparing his melting-point results with those of modern observers, I have been struck with the closenees of their agreement, especially when this is contrasted with the great discrepancy between modern results and those of Pouillet's predecessors.

Thus comparing the results of nine respectable pyrometricians who have, since Pouillet, determined the melting-points of pure gold and of pure silver independently, by different methods and under different conditions, I find that the greatest deviation from the mean is only 3 per cent in case of gold and 2.2 per cent in case of silver. Compare this modern accuracy with Wedgwood's determination of the melting-point of silver, 165 per cent above the mean of these nine observers; and even with Daniell's melting-point of gold, about 33 per cent above their mean. Yet, strange to say, these old and discredited melting-points and melting-points of other metals determined under the same false conditions, though they are no more fit to be set up against modern determinations than are the maps of the Chaldeans to be set up against those of a modern survey, still linger in well-known and reputable books.

But Pouillet's determinations of the melting-point of gold and silver (2192° and 1832° F. respectively) differ from the mean of these nine later investigators by only 12 per cent

* *Bulletin*, No. 54, United States Geological Survey, page 28, 1889.

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in case of gold, and by only 4 per cent in case of silver. In each case his results are above theirs.

Let the foregoing show that in advising abandoning Pouillet's data I am not ignorant of his worth. But, much as I respect his work, I am convinced that the temperatures which he has assigned to the different tints do not agree with common usage, be it because of his attempt to assign a special tint to each even hundred degrees of the Centigrade scale,* or because of some peculiarity of his eye, or because the names conventionally assigned in his country at that time to the color-impressions produced by different temperatures differed greatly from those now assigned to them here, or for whatever reason. I certainly have found my own color-impressions in reasonable agreement with those of other metallurgists; and on examining carefully my own color-impressions caused by definitely-known temperatures, I cannot force them into agreement with Pouillet's.

It is to be hoped that Messrs. White and Taylor will carry their work a little farther. They give a definite degree Fahrenheit corresponding to each color-name. While it is well to give such a temperature as the typical or specific one to which each name most precisely corresponds, will it not be well to give, in addition, the whole range of temperature to which each name may more generically apply? For instance, while 1975° is the temperature to which "Light yellow" most specifically applies, shall we not further give the boundaries between "Light yellow" and "yellow" on one hand and "White" on the other hand, saying "Light yellow," specifically 1975°, generically 1900° to 2050°, etc.?

It may not be amiss to touch on the question whether different substances give out different color-tints for one and the same temperature. I think that there is a general belief

† In proposing these color-names, Pouillet tried, not as might have been well, to assign to each color-name the temperature, or better, range of temperature to which it corresponds, but to assign a color-name to each even hundred degrees of the Centigrade scale; and this he did evidently with some misgiving, saying that he "believes that, without departing too far from the accepted meanings, we can make a distinct color-tint correspond to each hundred degrees." Thus his data should be taken as tentative; but the text-book writers have taken them as ex cathedra (*Comptes Rendus*, III, page 784, 1836).

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White and Taylor.			Pouillet.*				Howe.			
Name of color.	Temperature.		Name of color.	Temperature.		Excess + (or deficit −) over (or under) White & Taylor, deg. F.	Name of color.	Temperature.		Excess + or (deficit −) over (or under) White & Taylor, deg. F.
	C.	F.		C.	F.			C.	F.	
. . . . .			. . . . .			. . . . .	Lowest red visible in the dark . .	470	878	. . . . .
. . . . .			(Rouge naissant) Incipient red . .	525	977	. . . . .	Lowest red visible in daylight . .	475	887	−90 (under Pouillet)
Dark red, blood red, low red	566	1050	(Rouge sombre) Dark red . . .	700	1292	+242				
Dark cherry red	635	1175	(Cerise naissant) Incipient cherry red	800	1472	+297	Dull red . .	550 @625	1022 @1157	−28 @−18
Cherry, full red	746	1375	(Cerise) Cherry red . .	900	1652	+277	Full cherry . . .	700	1292	−83
Light cherry, bright cherry, light red	843	1550	(Cerise clair) Light cherry red (Orange foncé)	1000	1832	+282	Light red . . .	850	1562	+12
Orange	899	1650	Dark orange . . (Orange clair)	1100	2012	+362	. . . . .	. . .	. . .	. . . . .
Light orange	941	1725	Light orange . .	1200	2192	+467	. . . . .	. . .	. . .	. . . . .
Yellow	996	1825	. . . . .	. . .	. . .	. . . . .	Full yellow . .	950 @1000	1742 @1832	−83 @+ 7
Light yellow	1079	1975	. . . . .	. . .	. . .	. . . . .	Light yellow . .	1050	1922	−53
White	1205	2200	(Blanc) White	1300	2372	+172	White	1150	2102	−98
. . . . .			(Blanc éclatant) Brilliant white .	1400	2552	. . . . .	. . . . .	. . .	. . .	. . . . .
. . . . .			(Blanc éblouissant) Dazzling white	1500 @1600	2732 @2912	. . . . .	. . . . .	. . .	. . .	. . . . .

* *Comptes Rendus*, III, page 784, 1836.

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that they do, but that is a mistake. My reason for thinking so is the following:

We of course see objects only through differences either in the tint or intensity of the light reflected from them. Objects which emit the same tint and intensity of light cannot be distinguished from each other, no matter how different their texture, surface or shape may be. I have often, in operating on relatively small furnaces, brought the temperature at all parts of the heating chamber to very closely the same point. When this is done, all the different objects inside the furnace (fire-brick, sand, platinum, iron) after growing fainter and fainter, finally become absolutely invisible. The effect is often very striking. It is not at all due to the eye being dazzled, because this effect can be produced at a low yellow heat which does not dazzle the eye in the least. It is simply because every object, being at exactly the same temperature, emits the same tint and intensity of light; as all light from without is shut out by the enclosing walls of the furnace, the light which reaches the eye from objects within the furnace comes solely from their incandescence; and as the incandescence of all is exactly the same, they cease to be distinguishable from each other and from the furnace walls; or, in short, they become invisible. This phenomenon, which I have never seen described by any other observer, seems to me to show very clearly that these different substances must emit exactly the same tint for any one given temperature; for if they emitted different tints they would remain distinguishable from each other, or visible.

ON THE POSITION OF THE POINTS OF MAGNETIC TRANSFORMATION OF NICKEL STEELS.*

By L. DUMAS.

THE position of the points of the magnetic transformation of alloys of iron and nickel has been ascertained by Hopkinson, H. Le Chatelier, Guillaume, Osmond, and others. When the percentage of nickel is in the vicinity of

* *Comptes Rendus*, July 3, 1899.

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25° C., the temperature at which magnetism begins to appear, is not far from 0° C. If the nickel content diminishes (alloys called *irreversible* by Mr. Guillaume) or increases (reversible alloys), the temperature of the point of transformation increases rapidly.

We had been led to notice that nickel steel with the following composition: C 0.6 to 0.8 per cent, Mn 0.5, Ni 20 to 25 per cent, Cr 2 to 3 per cent, are not magnetic at the ordinary temperature, and remain so under the influence of cooling, even after immersion in liquid air.*

This fact was the starting point of our inquiry.† In the following table will be found the composition of our samples and their magnetic properties at the ordinary temperature, 15° C., in solid carbonic acid, —78° C., and, in some instances, in liquid air, —188° C.‡ The steels containing the same amount of nickel are grouped together, and in each group the samples are placed according to their carbon content:

	Chemical composition per cent.				Magnetism		
	Carbon	Silicon	Manganese	Nickel	at + 15°.	at −78°.	at −188°.
I.	1.37	0.41	2.71	10.00	very slight	very slight	permanent
II.	0.07	0.25	1.45	15.48	very strong	very strong	
	0.19	0.12	0.73	14.55	very strong	very strong	
	0.73	0.38	0.41	15.92	very slight	permanent	
	1.03	0.37	1.22	14.44	none	permanent	
	1.13	0.38	1.89	15.88	none	none	permanent
	1.36	0.55	2.07	14.80	none	none	
III.	0.15	0.15	0.31	24.06	very slight	permanent	
	0.34	0.22	0.51	24.04	very slight	permanent	
	0.41	0.23	0.11	24.05	none	permanent	
	0.64	0.27	0.88	24.61	none	none	not perman.
	0.85	0.47	1.41	23.35	none	none	
IV.	0.39	0.23	0.43	25.38	none	permanent	
	0.62	0.41	0.80	25.45	none	not permanent	
V.	0.23	0.17	0.18	27.12	none	permanent	
	0.25	0.14	0.36	27.72	very slight	not permanent	permanent

* Professor Dewar had the kindness to conduct this experiment in May and June 1897 with three samples; one of them underwent the magnetic transformation.

† The Commentry-Fourchambault Society entrusted us with this investigation, which was carried on at the Imphy Works.

‡ It is to the kindness of Mr. d'Arsonval that we are able to add to this table the very interesting results shown in the last column. We desire to express to him here our gratitude.

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	Chemical composition per cent.					Magnetism		
	Carbon	Silicon	Chromium	Manganese	Nickel	at + 15°.	at − 78°.	at − 188°.
VI.	0.36	0.47	14.4	0.60	0.9	very strong	very strong	very strong
VII.	0.52	0.24	2.83	0.59	4.95	very strong	very strong	
	1.14	0.24	3.12	0.93	5.05	very slight	very slight	
	2.13	0.54	3.92	5.05	4.96	none	none	
VIII.	1.77	0.40	3.19	2.78	7.28	none	none	none
IX.	0.31	0.42	2.92	0.46	10.20	very strong	very strong	
	0.73	0.58	2.70	0.61	12.04	none	permanent	
	1.10	0.34	3.55	0.91	13.34	none	none	none
X.	0.07	0.23	2.74	0.96	17.24	very strong	very strong	
	0.19	0.31	0.98	0.26	17.50	very strong	very strong	
	0.29	0.30	9.05	0.13	15.50	none	none	none
	0.33	0.21	2.83	0.60	15.08	none	permanent	
	0.40	0.47	1.75	0.63	16.06	none	permanent	
	0.50	0.24	1.77	0.53	16.68	none	permanent	
	0.53	0.35	3.02	0.82	16.05	none	none	none
	0.71	0.58	2.02	1.17	16.16	none	none	
XI.	0.10	0.21	0.43	0.32	21.84	very strong	very strong	
	0.27	0.23	0.56	0.45	22.08	very strong	very strong	
	0.28	0.29	0.59	0.36	23.06	none	permanent	
	0.30	0.23	0.53	0.27	23.73	none	permanent	
	0.31	0.25	5.29	0.23	24.20	none	none	not perman.
	0.45	0.35	0.28	0.66	23.26	none	none	permanent
XII.	0.63	0.94	2.53	0.41	27.16	very slight	not perman.	not perman.
				Iron.				
XIII.	0.11	0.23	9.87	0.70	1.65	none	none	not perman.
	0.46	0.52	9.40	1.72	1.75	none	none	not perman.
	0.96	0.49	9.62	1.45	1.45	none	none	not perman.

The examination of this table * leads us to the following conclusions:

1. The position of the point of magnetic transformation does not depend exclusively upon the amount of nickel present;

* Mr. Osmond, following up his remarkable work concerning the allotropic modification of iron, has experimented with liquid air. He called attention (*Comptes Rendus*, June 5, 1899, also *The Metallographist*, Vol. II, page 261) to two steels containing respectively 29.07 and 3.77 per cent of nickel, which became magnetic in liquid air. He has shown, moreover, that in austenite the carbon alone, by its own action, lowers the point of transformation of iron far below 0°C. His results are confirmed and generalized by our own observations.

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in each group the points of transformation are distributed over a range of temperature covering several hundred degrees C.

2. The point of transformation may in each group be lowered through the addition of carbon and manganese, resulting in steels which are non-magnetic at a low temperature, even with a very small proportion of nickel.

3. Certain steels containing over 24 per cent of nickel acquire after cooling some magnetism which is not permanent, i.e. which it looses at the ordinary temperature. (Mr. Guillaume's reversible steels.) Others, belonging to the same groups, assume through cooling some permanent magnetism (irreversible steels). One of the samples exhibit the remarkable property of being non-magnetic at $+15°$ C., of assuming some non-permanent magnetism at $-78°$, and some permanent magnetism at $-188°$.

4. The influence of carbon is very prominent, a few thousandths of that element being sufficient to lower the point of transformation to the neighborhood of $-188°$, while that of alloys of iron and nickel, which, moreover, always contain a little carbon, never falls below o° C.

The proportions of manganese are sufficiently small to neglect their influence compared to that of carbon and nickel. The manganese was introduced in the samples in order to promote the solution of the carbon and to avoid its precipitation in the graphitic state.

Chromium is a still more powerful dissolvent of carbon than manganese; it exerts a very favorable influence upon the ductility both in the cold and at a high temperature. It is for this reason that we prepared some samples of nickel steels containing various amounts of chromium.

The action of carbon remains prominent in most of the samples, but it appears to be null when the iron is displaced almost completely by nickel. On the other hand, chromium does not lower the point of transformation of steel containing a very small amount of nickel, but it lowers it very much if the metal contains large proportions of nickel, and especially when practically no iron is present. The magnetism acquired during cooling is also, as in the case of steel containing no chromium, either permanent or not. Four samples whose

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nickel content varies very much, but which are very rich in chromium, remained non-magnetic, even in liquid air.

The greatest lowering of the point of transformation was obtained through the combined action of carbon and chromium.

Constitution of Metallic Alloys. — Mr. Kerschkowitsch* sought to ascertain the constitution of several alloys by the method first used by Laurie, and which consists in measuring the difference of electro-motive force between a series of alloys containing various amounts of the two component metals, and that of one of these metals. When the composition of the alloy coincides with a definite compound, a sudden variation takes place in the value of the electro-motive force.

He did not find any definite compound in the case of alloys of cadmium and tin, cadmium and lead, zinc and tin, zinc and bismuth, copper and silver.

On the other hand he ascertained the presence of definite compounds in the following alloys: zinc-copper (Zn_2Cu), zinc-silver (Zn_4Ag), zinc-antimony ($ZnSb_2$), tin-copper ($SnCu_8$), and tin-silver ($SnAg_4$).

The Microstructure of Steel. — Mr. E. Heyn publishes, under the above title, an interesting article in *Stahl und Eisen* (August 1 and 15, 1899). He begins with some generalities concerning the microscopical examination of metals, describes the constituents of steel, and calls attention to the analogy which exists between metallic alloys and ordinary solutions, showing that steel at a high temperature should be considered like a solid solution of iron and the carbide Fe_3C.

In the following pages we reproduce in full the instructive photomicrographs which accompany Mr. Heyn's paper. A detailed and comprehensive description is appended to each figure.

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* *Zeitschr. f. Physikal. Chem.*, XXVII, 1898, page 123.

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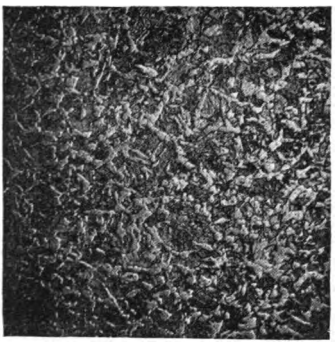


Fig. 1. *Hard Rail Steel.*
C 0.41 per cent, Mn 0.69 per cent.
Polished in relief. Magnified 90 diameters.
Depressed constituent: *Ferrite.*
Raised constituent: *Pearlyte.*

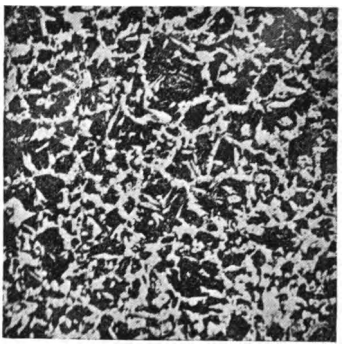
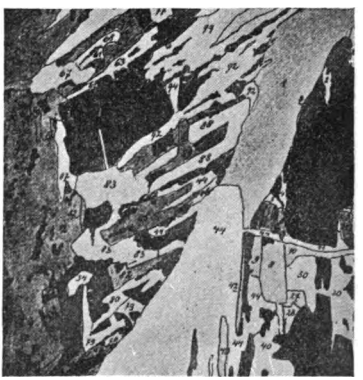


Fig. 2. *Same spot* as in Fig. 1.
Polished and etched.
Light depressed constituent: *Ferrite.*
Dark raised constituent: *Pearlyte.*



Figs. 3 and 4. *Soft Bessemer Basic Steel.* C 0.25 per cent. Section through ingot. Magnified 90 diameters. Hand drawings. Grains of ferrite of various sizes, some of them darkened through the action of chloride of copper and ammonium; small dark particles of pearlyte.

Fig. 3. *Center of Ingot.*
Grains are rounded.
Diameter of grains of ferrite: 5200 μ^2.

Fig. 4. *Outside of Ingot.*
The grains appear elongated.
Diameter of grains of ferrite: 5900 μ^2.

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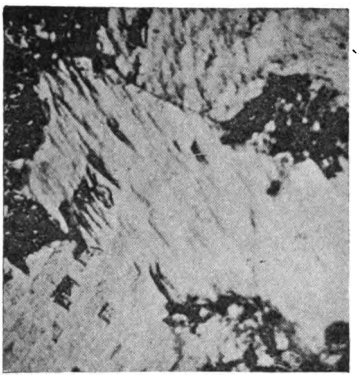
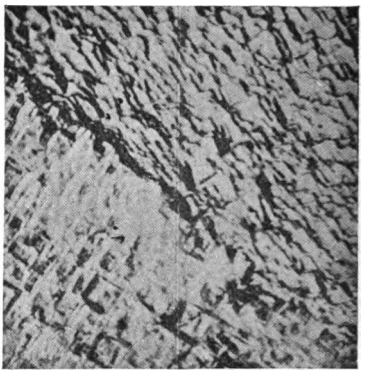
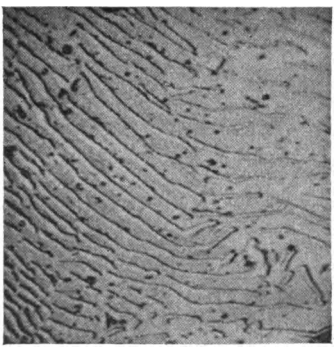




Figs. 5 and 6. *Soft Basic Bessemer Steel.* **Magnified 1240 diameters. Details of the structure of Fig. 3 under higher magnification. Appearance of a *single grain* of ferrite after etching with chloride of copper and ammonium.**

Fig. 5. Grain No. 30, 32 and 73 of Fig. 3.

Fig. 6. Grain No. 11, 10 and 2 of Fig. 3.



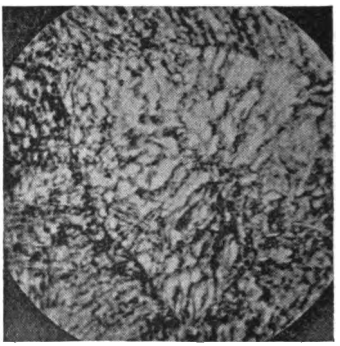


Fig. 7. *Pig Iron* **for the basic Bessemer process annealed in charcoal. Magnified 1240 diameters.** *Lamellar Pearlyte* **made up of hard thin plates of cementite alternating with wider plates of ferrite.**

Fig 8. *Granular Pearlyte.* **Magnified 1240 diameters. From a forged bar of crucible steel containing 0.92 per cent carbon. The whole mass consists of pearlyte.**

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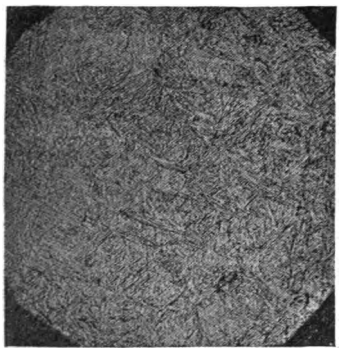


Fig. 9. *Tool Steel* from Böhler.
Marked Zäh. Hardened.
Magnified 270 diameters.
Etched with HCl in alcohol $\frac{1}{500}$.
Mass of *Martensite*.
Needles crossing each other.

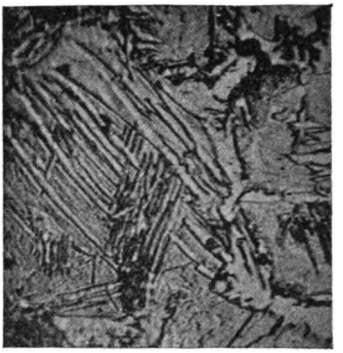
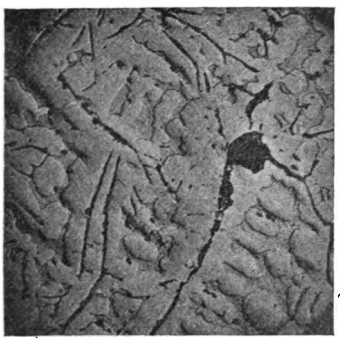


Fig. 10. *Soft Rail Steel.*
Quenched at 900°C.
Etched with HCl in alcohol $\frac{1}{500}$.
Mass of *Martensite*.
The needles are parallel to the three sides of a triangle.



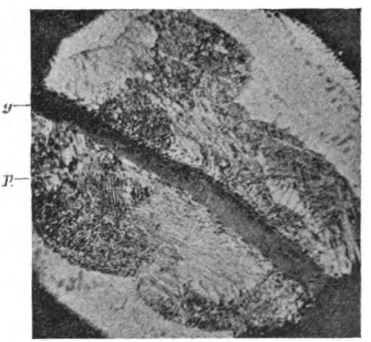


Fig. 11 and 12. *Grey Pig Iron.* Si 0.72 per cent, Mn 1.43 per cent, P 1.80 per cent.

Fig. 11. Polished in relief.
Magnified 90 diameters.
Raised rounded areas of *Cementite* surrounded by depressed *Pearlyte*, plates of *Graphite*.

Fig. 12. Polished and etched.
Magnified 675 diameters.
Showing cross section of a *Plate of Graphite* surrounded by pearlyte.

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Figs. 13–16. *Appearance of Pearlyte* as the carbon increases in unhardened alloys of iron and carbon containing less than 0.80 per cent carbon.

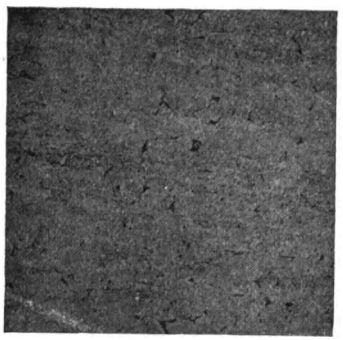

Fig. 13. *Soft Basic Weldable Steel.*
Plates for boilers. Polished and etched.
Magnified 90 diameters.
Background of colored ferrite with small dark particles of pearlyte.

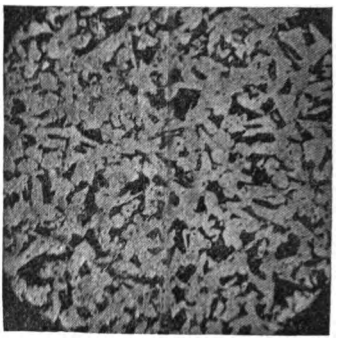

Fig. 14. *Medium Hard Steel.*
Rolled round bars. C 0.34 per cent, Mn 0.79 per cent. Polished and etched.
Magnified 90 diameters.
Background of ferrite and numerous particles of pearlyte.

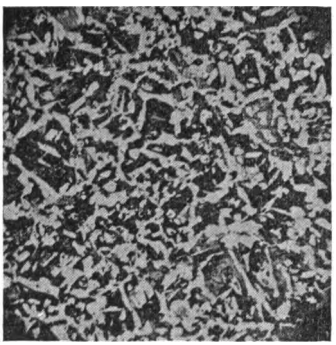

Fig. 15. *Hard Rail Steel.*
Rolled round bars. C 0.41 per cent, Mn 0.69 per cent. Polished and etched.
Magnified 90 diameters.
Large meshes of dark-colored pearlyte surrounded by a network of ferrite.

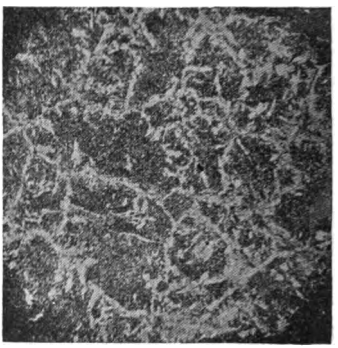

Fig. 16. *Open Hearth Steel Tires.*
C 0.50 per cent, Mn 0.72 per cent.
Polished and etched.
Magnified 90 diameters.
Very large dark meshes of pearlyte surrounded by a thin network of ferrite.

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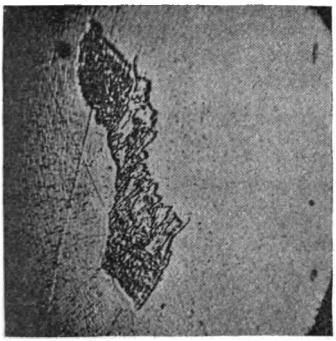

Fig. 17. *Soft Basic Weldable Steel.*
Same as in Fig. 13.
Magnified 680 diameters.
Enlarged image of a *single particle of Pearlyte.*
It consists of hard plates of cementite embedded in ferrite.

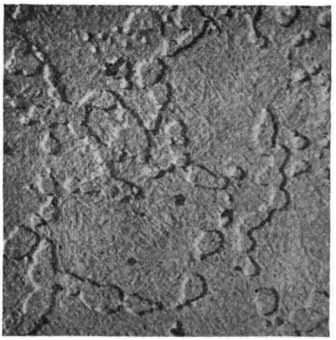

Fig. 18. *Styrian Steel.*
Containing over 1 per cent carbon.
Polished and etched.
Magnified 90 diameters.
Cementite stands clearly in relief. The other constituent is martensite (needle-like structure) which shows that the sample has been hardened.

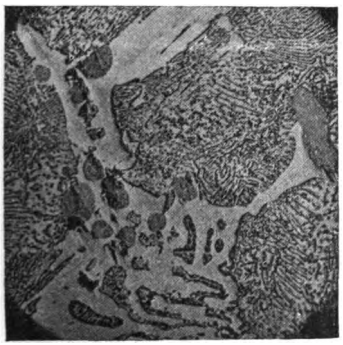

Fig. 19. *Pig Iron* for the basic Bessemer.
Annealed 108 hours in charcoal.
Polished in relief.
Magnified 270 diameters.
Cementite in relief. Pearlyte depressed.
Pearlyte has assumed through annealing the clearly lamellar structure illustrated in Fig. 7.

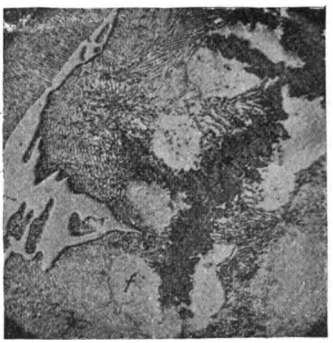

Fig. 20. *Same Sample* as Fig. 19.
Polished in relief.
Magnified 270 diameters.
The annealing caused the abnormal occurrence of ferrite and cementite side by side (some of the carbon of pearlyte was changed to temper-carbon).

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Figs. 21–24. *Böhler Tool Steel,* marked zäh. Hardened normally and at too high a temperature.

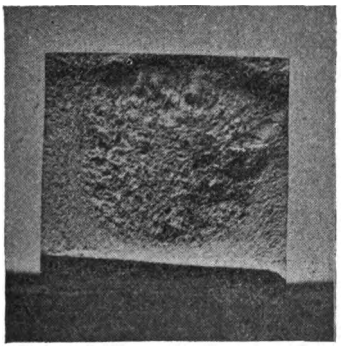


Fig. 21. *Normally hardened fracture.* Magnified ¾ diameter.

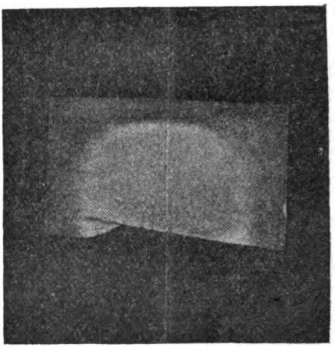
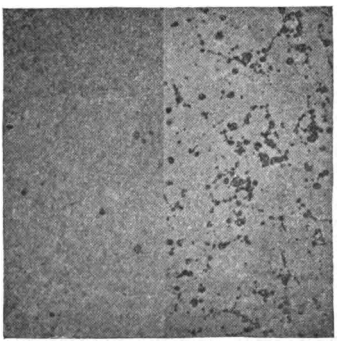
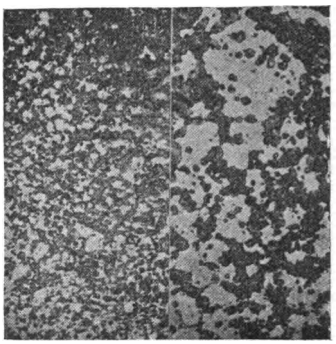


Fig. 22. Center of Fig. 21 polished and etched with chloride of copper and ammonium. Magnified ¾ diameter.





Figs. 23–24. Left halves of photographs show the structure of the normally hardened steel. The right halves that of steel hardened at too high a temperature. Etched with HCl in alcohol $\frac{1}{500}$. Bright constituent: Martensite. Dark constituent is of nearly the same hardness as Martensite. The structure is much coarser in the case of overheated steel.

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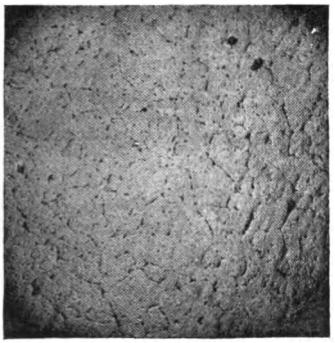
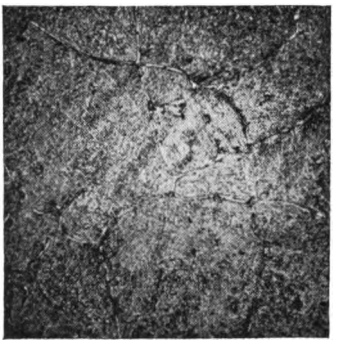
Figs. 25–30. *Soft Rail Steel* quenched at various temperatures. The steel of Figs. 25 and 26 was a rolled round bar (36 mm. diameter, and 50 mm. long). For the other experiments, cylinders 16 mm. long were used, cut from the center of the round bar, and heated and quenched when connected with a thermo couple. The samples were quenched immediately upon reaching the temperatures indicated. The heating took place in a muffle inside a porcelain tube, with the exception of the samples overheated.

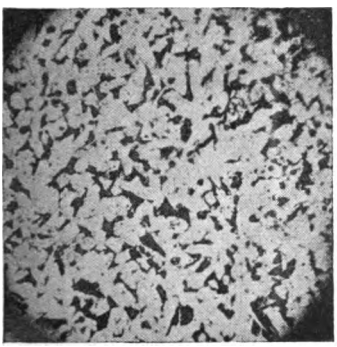




Figs. 25–26. Round bars heated in a forge until it showed signs of melting, and quenched in water.

Fig. 25. Polished in relief.
Magnified 22 diameters.
Slag-like dots on the border of the polyhedric grains.

Fig. 26. Polished and etched.
Magnified 90 diameters.
Bright bands forming a network around the yellow-colored martensite.



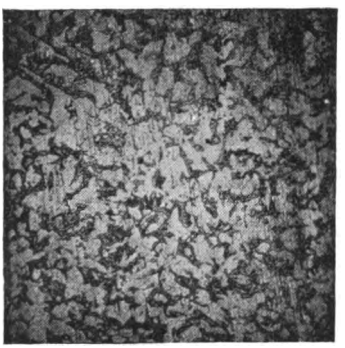


Fig. 27. Structure of the original round bar.
Polished and etched.
Bright ferrite and dark pearlyte.
Percentage of area of pearlyte, about 20.

Fig. 28. Small cylinder heated to 760° and quenched in water at 16° C.
Magnified 90 diameters.
Bright, ferrite, and a slightly raised constituent which under higher magnification reveals the characteristics of martensite.
Percentage of martensite in area, 22.

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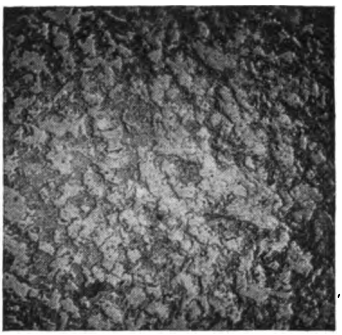

Fig. 29. Small cylinder heated to 800° and quenched in water at 15° C.
Magnified 90 diameters.
Bright ferrite,
and raised, yellowish martensite.
Percentage of area of martensite, 44.

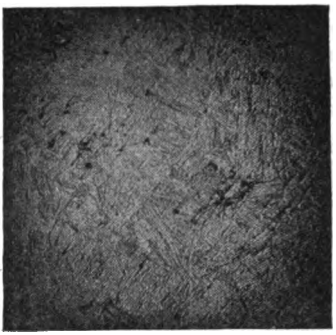

Fig. 30. Small cylinder heated to 900° and quenched in water.
Etched with HCl in alcohol $\frac{1}{500}$.
The whole mass is martensite.

Figs. 31–37. *Influence of the rate of cooling*, and of the temperature from which cooling occurs, upon the size of the cross-section of the grains of ferrite.
Rolled round bars of soft rail steel, 36 mm. diameter. C 0.21 per cent, Si 0.31 per cent, Mn 0.63 per cent, P 0.12 per cent, S 0.06 per cent.
Figs. 31–35 are drawings in which the ferrite is shown in white, the pearlyte in black.

Fig. 31. Rolled round bar.
Magnified 270 diameters.
Size of grains of ferrite: 500 μ^2.
Percentage of area of pearlyte: 19
$\mu = 0.001$ mm.

Fig. 32. Small cylinder of same steel.
Heated to 1000° in muffle and quickly cooled in air upon an iron plate.
Time of cooling from 1000° to 400°: 2 to 3 minutes. Size of ferrite grains, 530 μ^2.
Percentage of area of pearlyte, 16.

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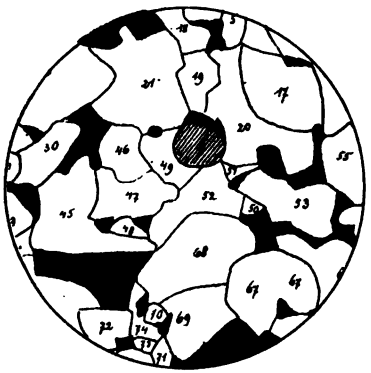

Fig. 33. Small cylinder of same steel.
Heated to 1000° and slowly cooled.
Time of cooling from 1000° to 400°: 31 minutes.
Size of ferrite grains, 775 μ^2.
Percentage of pearlyte in area, 18. S = slag.

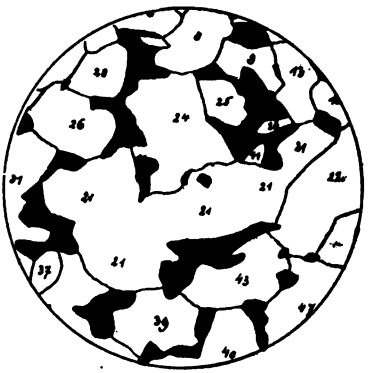

Fig. 34. Same steel. Round bar.
Heated to 1000° and cooled slowly.
Time of cooling from 1000° to 400°: 73 minutes.
Size of grains of ferrite, 1100 μ^2.
Percentage of area of pearlyte, 19.

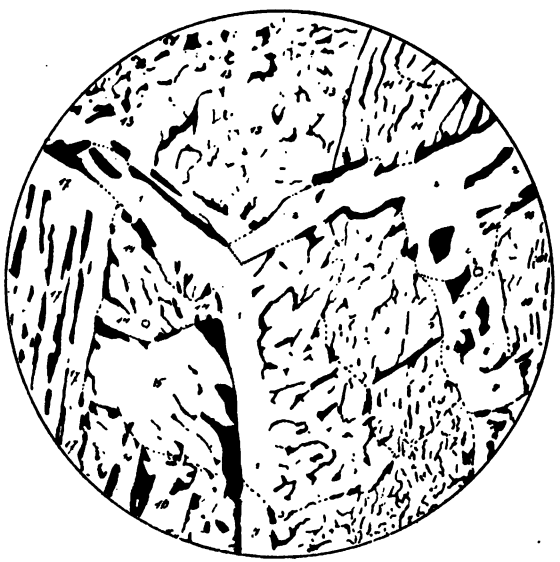

Fig. 35. Same steel heated in a forge until it began to melt,
and allowed to cool in air.
Size of grains of ferrite: about 5400 μ^2.
The pearlyte follows no longer the outside of the ferrite grains,
but is disseminated through the grains.

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Fig. 36. Same steel and same treatment as in Fig 35.
Polished and etched.
Magnified 22 diameters.
Network of ferrite and meshes of mixture of ferrite and pearlyte.

Fig. 37. Same steel and same treatment as in Figs. 35 and 36.
Higher magnification (90 diameters).

Figs. 38 and 39. *Influence of work* at the ordinary temperatures upon the structure of soft steel.

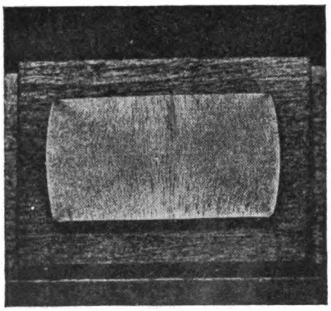

Fig. 38. ½ natural size.
Compression-test. Etched with chloride of copper and ammonium.
Two bright cones with the compressed surfaces as bases.
Two dark cones in the opposite directions.

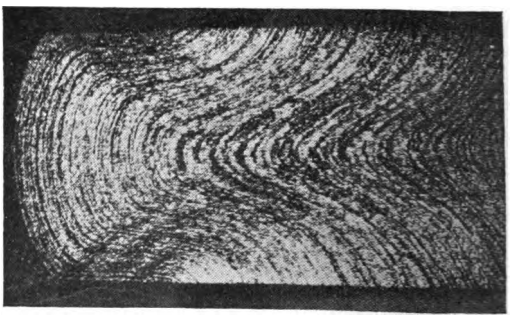

Fig. 39. Hammer-test after five blows of 20 mm.
Rolled soft steel.
Magnified 7 diameters.

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Figs. 40–42. ***Segregation in a Cast Ingot*** **of soft basic steel. Carbon 0.25 per cent.**

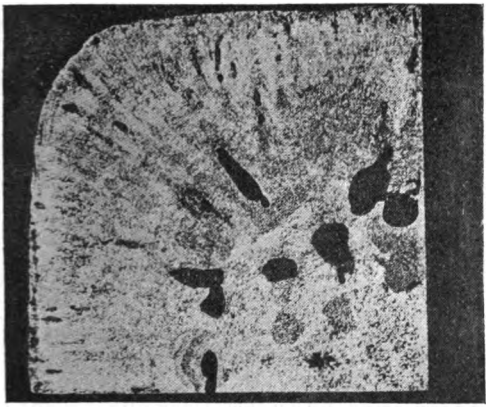

Fig. 40. Section through ingot. Natural size.
Etched with chloride of copper and ammonium.
Dark deep cavities: blow-holes.
Grey: spots around the blow-holes.

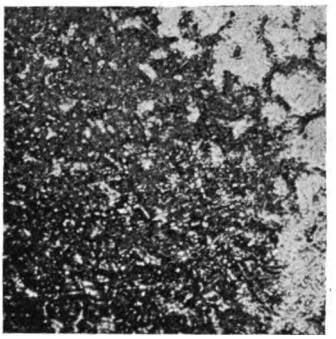

Fig. 41. One grey spot. (See Fig. 40.)
Magnified 21 diameters.

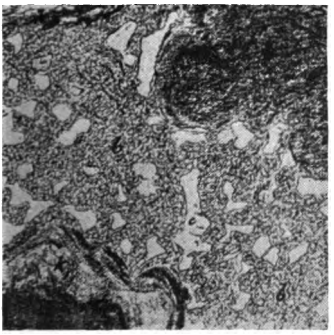

Fig. 42. Hand drawing.
Magnified 270 diameters.
Segregation around a blow-hole.

Fig. 43. Section through piled and forged wrought iron.
Magnified 1.1 diameters.
Polished in relief.

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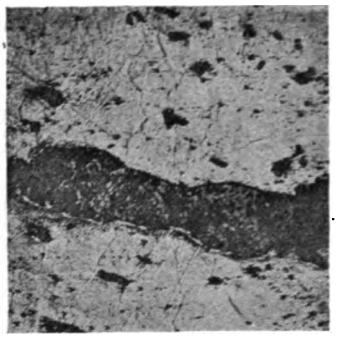

Fig. 44. Section through coarse-grained puddled iron.
Polished and etched.
Magnified 90 diameters.
In the dark slag bright dots are to be seen.

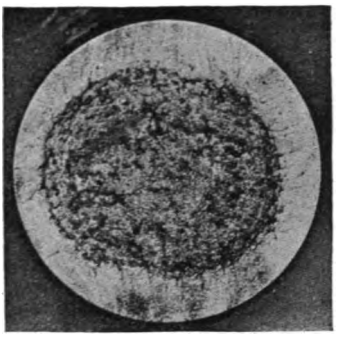

Fig. 45. Axle of soft steel.
Etched with chloride of copper and ammonium.

Practical Microscopic Analysis for Use in the Steel Industries. — Mr. C. H. Risdale read a paper of some fifty pages at the August 1899 meeting of the Iron and Steel Institute, entitled "Practical Microscopic Analysis for Use in the Steel Industries, with an Introduction to a Systematic Study of Soft and Dead Soft Steel," the most important features of which are reproduced in the following pages:

Some of the Points Previously Verified.

1. That good normal steel, or steel of the very purest quality, which was heated for a *short time* (three or five minutes) to a *high temperature* (near to welding but not to burning) and allowed to cool very slowly, developed large grains and became very brittle, or more properly *weak* or *rotten*, breaking generally with the first blow, with little or no deflection. The grains were generally so large that microphotographs were unnecessary.

2. That the same steel treated in the same way throughout, except that it was *chilled*, showed almost the same degree

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of weakness, the chilling not having been able to sufficiently break up the structure to restore toughness.

3. That the same steel heated for a *long time* (say 3½ hours) at a quite *normal* and what is generally considered *harmless* re-heating temperature,* and either allowed to *cool slowly* or *chilled*, showed the same properties.

4. That re-heating to cherry redness (900° C. or over), whether allowed to cool slowly or chilled, restored the toughness in steel which had become brittle from any of the above causes. Special stress was laid on the re-heating being *rapid*. †

5. That the restoration of toughness was *due to the breaking up of the grain* (Figs. 30, 31), also billet (Fig. 29) when rolled to wire rods (Figs. 32, 33a, 33b), and when forged to bars (Figs. 26 and 27).

6. That steel of the same quality became very brittle if worked at a blue heat.

7. That re-heating restored the toughness in steel rendered brittle by this means also.

Classification — Soft Steel.

Class I. *Steel of normal treatment and normal composition*, i.e. steel which has stood the normal processes satisfactorily and has given the normal commercial results, such as softness, toughness, soundness, or other qualities.

Class II. Steel of *normal* treatment but *abnormal* composition, results known.

Class III. Steel of *known abnormal* treatment and normal composition, results known.

---

* Mr. Stead has shown in his paper read in May 1898 that similar changes, as far as the grain is concerned, but not accompanied by as much brittleness, are produced by very long heating (say 48 hours) at a low red temperature.

† He has also shown, in his paper given in autumn of the same year, that (with more or less uncertainty) breaking up of the grain and corresponding restoration of toughness is brought about by slow re-heating below 900° (say about 700°, viz. re-annealing). — *Journal*, 1898, No. II, page 147, particularly last sentence.

Mr. Stead's two papers have been published in *The Metallographist* (October 1898 and April 1899).

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(*a*) "*Soaked*" *longer than usual* at *normal* temperature, and treated afterwards in various ways.

(*b*) Heated to *too high* temperature for *short* time, and *no* work, or work finished at too high a temperature.

(*c*) Heated to *too high* temperature for *short time*, but worked down to normal finishing temperature — low red heat.

(*d*) Work continued to too low a temperature (as blue-heat or cold).

Class IV. Steel of *known abnormal treatment* and *abnormal* composition, results known.

Class V. Steel of known *abnormal results*, treatment unknown.

(*a*) "Dry" opens at ends in rolling.

(*b*) "Red-short," ragged edges.

(*c*) "Hard," poor elongation, twists, or bend tests, and high tensile strain.

(*d*) *Cold-short* or brittle, to a moderate extent.

(*e*) "*Rotten*," "breaks like pot."

(*f*) *Hollow*, seamy or laminated.

(*g*) *Blistered.*

Medium and hard steel can follow on exactly the same lines.

Size of Grain.

It may prove a definite rule that in normal steel, for sections of such a size that they are finished below the critical temperature* but not below low red, under similar heat conditions (i.e. finished at the same or *pro rata* temperature and subjected to the same cooling influences), in steel of the same composition, the size of the grain is *inversely proportional to the sectional area*, and when it is not so this gives us an indication of the variation in the other conditions. So far, all the measurements taken closely confirm this, in the smaller sizes the influence of decreased finishing temperature being more marked.

Therefore the *size of grain* in any piece of steel in relation

* The temperature above which work does not reduce the size of grain, as described by Brinell.

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to the sectional area of the piece is of very considerable importance.

It may be well to state here that by size of grain is meant the average "maximum size" of grain or the average size of grain of which a full sectional area is presented. A great many of what appear to be grains are only sections of *one part or corner* of the grains, and the more homogeneous and broken up the structure is the more this is so, whilst the more completely the grain has been able to form (or crystallize out) and the more regular it is, the larger and better defined the sectional areas are.

RESULTS OBTAINED WITH LOW CARBON STEELS. — The following are some of the conclusions arrived at from the examination of a very large number of samples, and for which the evidence so far seems to be sufficient to warrant the indication given being put forward tentatively as definite tests, though it is to be hoped other workers will follow up the investigation and establish them more completely, or modify them as may be required.

Class I. *Normal steel.* — Steel in which work has been continued to below critical temperature, viz. to from good red to low red. See also definition already given previously.

Shows what may be termed an "uninteresting" or regular structure which is almost homogeneous, the grains *small and often ill-defined.* The grains at the outer edge of the piece are at least as small and frequently *smaller* than at the centre, owing to the work on the outside. This is an important feature. They continue right up to the edge.

Class II. *Steel of normal treatment but abnormal composition: Results known.* — Ordinary commercial soft steel only has been dealt with in which the carbon certainly will not exceed 0.20 per cent, nor the manganese 0.60 per cent, and which, if they contained as much as 0.10 per cent of either silicon, sulphur, or phosphorus, would be regarded as of abnormal composition. Within these limits the grain size is *practically unaffected.* Indeed, even with phosphorus (which particularly tends to give large grain), there is only a slight effect. It therefore may be safely concluded, should the grain differ much from that in normal steel, even though the composition is "abnormal" within the limits stated, that this must be at-

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tributed to other causes, such as the treatment whilst working, and *not* to the slightly increased impurities.

Class III, *a*, *b*, *c*. *Steel that has been heated too long or to too high temperature*, or finished too hot, but without *burning*, shows, according to the extent and manner in which this has been done, either an outer "carbonless" ring, or a ring of large grains, the grains being *larger* at the outside than further in, which is the reverse of what is found in normal steel, and is a sure sign of the above treatment.

This carbonless ring is often, with light etching, plainer to the eye than by the aid of the microscope. Sometimes, generally with depper etching, it appears to the eye as a dark band.

At the edge sometimes the grains are quite separated, the junctions being wide open. Then it is approaching to "burnt" steel.

Class III, *d*. *Steel on which work has been continued to too low a temperature.*

(*a*) To blue heat, or through blue to straw.

(*b*) Steel worked cold.

(*a*) *Steel worked at or through blue heat.* — In order to properly understand what to look for, let us consider what takes place when steel is worked at various temperatures.

If worked only *above critical temperatures*, which for soft steel is from good yellow to white or welding heat, and allowed to *cool slowly*, the size of grain is affected very little or not at all by the work, and when cold, a section shows no flow-lines or signs of deformation. If *chilled sufficiently rapidly*, such signs may be seen. For instance, a cross, in a small bar forged to low yellow heat and then suddenly chilled. In the first case, slow cooling from a high temperature allowed grains to arrange themselves and flow-lines to dissipate. The material being quite plastic at the temperature at which worked, the grains could move freely, hence their contact is good and the junctions sound; but if chilled before they have re-arranged themselves, signs of the flow are left.

If worked to *below* the *critical* (viz. to a *normal*) *temperature*, say good red to low red, the grain becomes smaller, and well interlocked or homogeneous, and when cold, flow-lines are more or less distinct, according to the temperature,

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mass, and rate of cooling. In this case, too, the material being plastic at the temperature at which worked, the grain contact is good and their junctions sound.

If worked through or to blue heat, or subjected to shocks at this temperature, the effect tends in the same direction, but meets with a new limitation.

The material at this temperature appears to be in its condition of *minimum plasticity*.

If the blow or work is too light to distort the piece, it simply *sets up a state of strain* at the surface, which penetrates more or less deeply, according to the force exerted.

Provided there is no actual distortion, however near the force has come to producing it, there can be no evidence visible through the microscope. The same will be true provided the distortion is not sufficient to yield flow-lines which can be distinctly recognized, although there may be a considerable state of internal strain or predisposition to rupture.

When the force is more powerful, notable distortion takes place, and its evidences, which take many varied forms, are clearly revealed by the microscope.

According to the nature of the work, whether light and rapid, as in hand-hammering, heavier and less rapid, as in steam-hammering (the work in these cases being shock), or whether strong and steadily applied, as in rolling, and according also to the mass of the piece subjected to it, so the visible effects vary, both in character and in the extent to which they penetrate the mass or are confined to the surface.

We have so far noticed the following indications, either or all of which may be present, and which are best seen by vertical illumination: —

1. Breaking up of the grain at the surface, penetrating more or less deeply.

2. Loosening of the grain or opening of the granular junctions, and frequently actual rupture.

3. Flow-lines in various directions from the part where the force was applied, marking the direction in which the material has slipped, and indicating lines of weakness, the portions which have slipped being also in a strained or disturbed condition, these flow-lines culminating in

4. Cracks of all degrees.

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Apart from the local indications 1 and 2, there is no special size of grain associated with steel which has had work at a blue heat, this being determined, as usual, by the mass of the piece and its previous history.

If the grain has been large before, it is still large except at the parts where work has broken it up, if it has taken effect in this way. This is the case if a piece of steel has been heated to above critical temperature, say to good yellow heat, and allowed to cool slowly without work on it till below red heat, and explains the increased brittleness found under these conditions and described by the author, page 225, in the paper on "Brittleness in Soft Steel," read before this Institute, May 1898, the reason being, of course, that the grains, and hence the continuous junctions, are larger.

The flow-lines in square bars are frequently at an angle of 30° to 45° to the surface, and in such cases often take the form of a more or less regular and complete cross which may have straight or curved arms — apparently the harder the material at the time of forging, the straighter the arms of the crosses, whilst if softer they are more curved, also those in hand-hammered bars being less regular, and those in steam-hammered bars more regular, being generally more marked in proportion to the degree to which brittleness has been developed. In other shaped pieces the flow-lines are not so distinct or regular. The flow-lines, though sometimes visible before etching, are brought up much more clearly by it. They are frequently visible to the naked eye as dark markings on the surface. They are no doubt apparent on account of the different orientation of the crystals in the portions which have slipped, as compared with that of the adjoining portions. If by chance the orientation were the same in the adjoining portions, the flow-lines would be no longer apparent, which may perhaps account for the imperfection of parts of the crosses in certain cases.

Anton R. von Dormus* mentions some crosses visible to the naked eye in forged bars of hard (chisel) steel. He attributes the different appearances in that case to a difference

* *Weitere Studien über Schienenstahl.* Anton R. von Dormus, Ingenieur der Kaiser Ferdinand-Nordbahn. Oesterr. Ingenieur- und Architekten-Verein. Wien, March 26, 1898.

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between the carbon of the inner and outer portions, and reference is only made to them as bearing on the direction of flow in forged bars. Flow-lines, whether in the form of a cross or otherwise, are *not* in themselves a distinctive indication of work at a blue heat.

To sum up the causes of "blue-heat brittleness," there is no evidence so far of it being due to symmetrical arrangement of the crystals, or, indeed, to any particular structure *per se*, but to *strains*, and more or less complete *rupture* of a number of individual granular junctions or cleavages, developing sometimes into continuous cracks, all being due to *distortion* produced by work when the material is in its least plastic condition, i.e. at that particular temperature when, owing to its hard or brittle state, work is able to produce these effects.

Indeed, microscopic examination and other evidence brings out clearly the *vital fact* that whilst at a blue heat steel is in a condition of peculiar receptivity or sensitiveness to *retain* in permanent form (strains, cracks, etc.) the effects of shock or work. No doubt this retention of the effects (besides actual cracks formed at the time, which no subsequent treatment could remove) is largely due to there not being sufficient heat for the parts which are merely strained to re-adjust themselves whilst cooling, as is the case when work is finished at a red heat, and still more so when finished at a yellow heat.

There can be no question as to the brittleness being *permanent*. Samples prepared eleven years ago, viz. in 1888, were recently broken, and were still as brittle as ever.

As further showing that the brittleness is not necessary due merely to *surface* distortion or to cracks *actually developed*, but also to *state of strain* and *hard condition* which has equally been retained, a sample from which the outer $\frac{1}{16}$ inch was filed (thus removing any surface cracks), though it did not break without deflection as before, broke on bending through 40°, and was therefore not tough. The crosses going right through the pieces also show that the effects are more than superficial, whilst the cutting, filing, grinding, and polishing give abundant proof of the *greater hardness* right through.

The practical question as to what amount of shock or

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work at blue heat is necessary to cause brittleness is one which cannot be answered exactly, but there have been plenty of instances where in the case of plates the work and conditions they have gone through, together with subsequent vibration or shock, have been sufficient to cause fracture.

The question *why* steel is peculiarly sensitive at these temperatures still remains unanswered. But however interesting it might be to pursue the inquiry, it is not of *practical* importance, it has not incidentally been revealed by the microscopic examination, and it is outside the scope of this paper.

(*b*) *Steel worked cold.* — The same general features are presented as in steel worked at blue heat, but for the same amount of work the effects of distortion are not apparent to so great an extent owing to the greater plasticity of the material at that temperature.

Provided the work is sufficiently severe, almost identical effect may be produced though there is not so much of the appearance of "loosening" of the individual grains.

The blue-heat worked bars were not perceptibly reduced in size, and the hammering was relatively light; even when steam-hammered, the blows were as light as possible. The bar which was cold hammered from $\frac{3}{4}$ inch square to $\frac{1}{2}$ inch square presents an appearance almost identical to a blue-heat hammered bar.

The flow in a cold sheared plate is also clearly visible, and shows the pull.

Attention may here be drawn to a point of considerable practical importance which is by no means as widely recognized as it should be, viz. the *undoubted fact* that both surface cracks and hollowness in the mass can be developed in material at points where it was before *perfectly sound*, by work at too low a temperature.

This seems to be particularly the case with *forging*, where the work is applied in a succession of local shocks repeated at certain points, thus producing an accentuated flow or creep along corresponding lines, and a more or less symmetrical disposition of material. Indeed the Mannesmann Tube Process depends entirely for its success on the readiness with which, under certain conditions, even in hot

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material unsoundness can be produced in the centre of sound steel.

Note also the increased tendency to lamination in plates and sheets, and to blistering in tin plates and cold rolled bright strips, even the "hot" work on which is often very near blue heat, and which also often undergo further cold rolling.

There can be no doubt that the surface and interior do not spread equally, the difference at times being so great as to cause rupture along the lines of flow, which is frequently revealed on pickling.

The author is quite aware that it is usual to attribute this wholly to blow-holes, but it does not follow that because certain sheets are sound, the steel from which they were made had no blow-holes. Blow-holes exist to a greater or less extent in *all* steel of *every make*, but the work the material undergoes closes them up, and it depends very largely on the conditions of further working whether or not they are reopened and manifested. What it is here sought to point out is that in a great number of instances where blisters appear these are *due to variations from the normal conditions, in the temperature of working up, in pickling*, etc., and it is these which have brought about an actual separation of the fibre which would not otherwise have occurred.

The fact remains that the same steel, which in tin plates, etc., would give blisters, in sections of greater mass (work on which is consequently finished at a higher temperature) does *not* yield blisters when pickled. Either a billet, or for that matter a blistered wire rod, would be a curiosity worthy of sending to the British Museum.

The fact is, distortion strains exist in *all* worked material at the time of working (though of course they are less the softer the material at the time of working), but when worked hot they are dissipated by slow cooling, and in most cases more or less fixed by chilling. They are also dissipated by re-heating.

The same general characteristics as have been described for cold-worked soft steel, viz. the flow following the lines of force, have already been shown for hard steel by Mr. Stead*

* *West of Scotland Institute*, No. I, Vol. iv, November 1896, "Micro-Mechanical Examination of Old Steel Rails and Tyres."

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in his now well-known photographs of the effect of wear on a rail, tyre, and chisel. The causes are the same as in cold and blue-heat-worked soft steel: work causing compression of the surface, hence flow, hence weakened cohesion of the mass affected.

Although there is perhaps, in the absence of knowledge as to the amount of work which a sample has undergone, no very distinct indication as to whether brittleness has been produced by work at blue heat or work whilst cold, in practice this is hardly likely to prove a difficulty, as the conditions of manufacture will generally be sufficiently well known, or even apparent from the piece itself.

Indeed, in seeking for the cause of brittleness in any particular instance, the evidence which the microscope affords as to blue or cold working may be rather of a negative character, viz. the *absence* of evidence of excessive or prolonged *heating*.

If the sample is thin and the brittleness not due to composition, weld-heating, soaking, or excessive oxidation, then it is necessarily due to blue or cold working, and which of these, as already stated, can generally be distinguished.

Class IV. *Steel of known abnormal treatment, and abnormal composition, results known.* — Just as Class II in the main closely resembles Class I, so Class IV, broadly speaking, resembles Class III, only as fresh factors are introduced the results obtained are correspondingly varied.

Class V. *Steel of known abnormal results, treatment unknown.* — Steel of this class should always be analyzed, and its composition borne in mind when examining it microscopically, and special endeavor should be made to procure suitable standards, as referred to already.

The different features of one or other of the previous classes will no doubt be met with and readily identified.

(*a*) and (*b*). If the steel on analysis proves to have been of suitable composition for the purpose for which it has been used, faults of this class generally prove to be varieties of *burning*, but it would take too long to deal with this here, and as a good deal has already been published on the subject, and a number of examples shown by other writers, it is not proposed here to go into it to any length. The ordinary in-

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dications are frequently sufficiently unmistakable without the aid of a microscope, and it is generally only needed to determine whether, in the cases of certain sections, tears and cracks are due to pull of the rolls through improper draughting or feeding, or to red-shortness. The characteristics are those of Class III, (*a*), (*b*), (*c*), but intensified, according to the extent burnt. There is the carbonless ring or band along the edge penetrating more or less deeply, accompanied by oxide cracks and fissures between the grains, these latter generally being quite plain when the section is examined in the polished state before etching.

(*c*), (*d*) and (*e*) generally prove to be due to the same causes as in Class III, (*a*), (*b*), (*c*) or (*d*), if steel has been of suitable composition for the purpose used.

(*f*) and (*g*) are mechanical defects which may easily be recognized, and the accompanying structure generally shows quite clearly whether these have been initial defects present in the steel when supplied, or whether there has been anything in the subsequent treatment of the steel to account for them.

In conclusion, the whole object of the investigation described in the present paper has been to obtain information, deductions from which shall be of *practical* use, such as data either

(*a*) To *definitely* confirm what are generally accepted as correct conclusions, but for which such definite data were not published.

(*b*) To show how certain behavior under mechanical or physical treatment is brought about, and hence, by inference, how to avoid such behavior if objectionable.

(*c*) As to the indications that steel is likely to show certain behavior, beyond what can be judged by composition.

The time has now arrived when it should be well recognized that composition only indicates such well-defined effects as are generally understood *without certain narrow limits* of treatment, which are termed "*normal.*" The moment we get outside these limits, the effect of the *treatment* far outweighs that of the *composition*. Often so-called "normal" treatment in manufacturing processes is really abnormal, i.e. the effect of the treatment has outweighed that of the composition.

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Makers of steel can only control the composition, which is often the least powerful factor, so it is most important that, since competition frequently leads users to more or less abnormal or unsuitable treatment, they should understand what it does.

Whilst it is objectionable to dogmatize — and in advancing any views at all there is always a risk of being afterwards proved to have drawn wrong conclusions, especially if a subject be looked at too narrowly, as exceptions can always be found — still, if progress is to be made, definite statements must be formulated as starting-points.

DETAILS OF PRACTICAL MANIPULATION. *Sampling.* — It is not practicable to section the whole area of large pieces, say over $\frac{5}{8}$ inch square, and in most cases a total area of $\frac{1}{4}$ inch to $\frac{3}{8}$ inch square is preferable. It is, therefore, important to follow a fixed rule, and always cut the section from one definite position, so as to be of value for comparision. This is more obvious, as the grain may vary a good deal from the center to the outside in section of large area.* The section cut can, of course, be a *cross*-section, or one parallel with the direction of rolling, or a section of the outside surface. For many purposes, including grain measurement, we prefer the cross-section, and this may be sufficient, though in some cases the other sections are desirable.

In *billets* half the section, $\frac{3}{16}$ inch thick, is sliced off. Then the strip AB is cut, forming a section from opposite sides right across the center; this is again cut in half, each half showing all the variations from centre to outside, and the centre of each half, i.e. the part midway between the centre and outside of the billet, is perhaps most truly representative of the whole. Strip A is polished on the face shown, thus being a *cross-section*. Strip B is polished so as to give a section in the direction of rolling or *side elevation* of the section, and if desired C can be cut and polished to show a plan of the *surface*.

For *flats* the position most truly representative of the mass is along the line midway between the centre and outside as D, but whether the cross-section be taken at this point or

* Mr. Albert Sauveur drew attention to this in the case of rails in his paper before the American Institute of Mining Engineers, August 1893.

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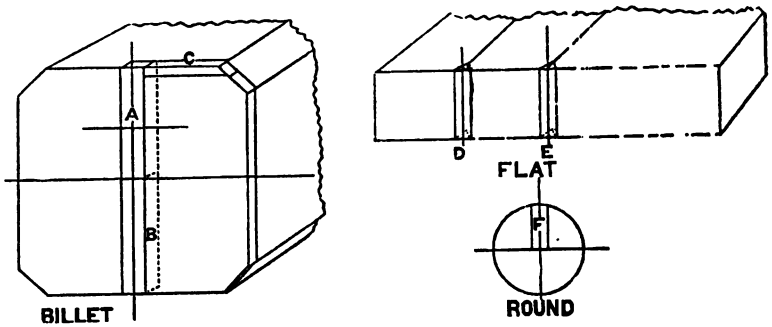
along the centre line as E, a V-shaped cut, having a face $\frac{3}{16}$ inch broad, will in most cases be sufficient, and less trouble than cutting off a full slice. The two ends of the section so cut will represent two outer sides.

From *rounds*, if only about $\frac{3}{8}$ inch diameter, a full slice is cut, but if larger than this, a half section is cut and then the piece marked F cut out of it.



For very small sections, as wire, thin sheets, etc., it is convenient to fix a number together by forcing them tightly into a piece of bicycle or other steel tube, or binding with tin sheet, and filling up the interstices with fusible metal such as melts at below the boiling point of water — which low heat insures that there shall be no interference with the structure. A section is then sawn off the whole.

Sections are never cut too near to any distorted part, such as a sheared edge or fracture, since the grain arrangement is not normal in such a case.

Grinding. — This is done substantially as already described by Mr. Stead and others. For most purposes we give the following treatment: —

Rough filing.
Fine filing.
Rough commercial emery cloth stretched on a board.
Fine emery cloth stretched on a board.
Finer specially prepared emery paper on disc in Mr. Stead's polishing machine.
Diamantine on cloth on disc in Mr. Stead's polishing machine.

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Rouge on wash-leather on disc in Mr. Stead's polishing machine.

Rouge on wetted parchment stretched on glass for very fine work. For most purposes exceedingly fine polish free from all scratches is not necessary.

Mounting. — The sections are mounted by means of a microtome, with a very little ordinary modelling material (a sort of grease and clay mixture which always remains plastic).*

Protecting. — If now or at any later stage the surface requires protection from corrosion, a drop of solution of resin in benzol is put on it. This, when required, can be at once removed by benzol and a clean soft rag.* Definition of grains, especially by oblique illumination, is often lost on removal of this, but it is the best method we know of so far. In spite of this, if samples are kept in a place that is at all damp, streaks of rust work under it.

Etching. — For determination of size and definition of the grain, a solution of iodine in alcohol is generally most suitable, and the operation can be conducted under the microscope, and being watched whilst in progress, can be judged to a nicety. Usually one to two minutes is sufficient. The section is then washed with a few drops of methylated spirit, and generally rubbed with the finger to avoid spurious markings, then pure alcohol, and dried by rapid motion or heat; and generally it is rubbed with wash-leather. When finished it is so light that usually it can hardly be seen with the naked eye.

Etching with acid, on account of the bubbles and acid spray and vapors given off, is far more likely to injure the reflector, so this is not generally done under the microscope. Sections are then washed with water, lime water, water again, methylated spirit, and pure alcohol.

Care must in all cases be taken that the etching fluid evenly and simultaneously covers the whole area of the section, otherwise various markings and other differences

* Method suggested to us by Mr. Stead. See also his two papers, *Journal of the Iron and Steel Institute*, 1894, No. I, "Methods of Preparing Polished Surfaces on Iron and Steel for Micro-Examination"; 1897, No. I, "Micro Accessories for Metallographers."

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may result, which will give rise to misleading conclusions, especially as the relative rate of attack is sometimes of considerable significance.

Illumination. — For moderate powers, such as can be obtained from a 1-inch objective, oblique illumination with Sorby's parabolic reflector gives a more general and detailed, or what may be termed "perspective" view, and in most cases is useful for grain measurements, showing the grain outlines sufficiently clearly.

Sorby's 45° reflector gives a "plan" or view of the grain outlines, illumination being vertical, and in some cases is preferable. For instance, flow lines which are scarcely noticeable under oblique illumination may be easily apparent with this.

In both the above cases it is better to have previously removed the glass disc of the Beck's illuminator, if this is also fitted on the microscope.

Beck's illuminator is chiefly useful for higher powers, as ¼-inch and ⅛-inch objectives — for which it is indispensable — with 1-inch objectives, and a low-power eyepiece, as No. 1 or 2; this latter form of illuminator does not give a full field. It gives vertical illumination.

An incandescent gaslight, with a reflector behind and condensers in front, will be found a most satisfactory source of light.

Examination. — Just as care is taken that the section cut shall represent as nearly as possible the whole of the piece from which it is cut, so equal care should be exercised not only that the whole section cut should be examined, but that the part of it selected for the photograph should be *strictly representative* of it.

It is important to keep this ever before one's mind, and not to be tempted into keeping as a record a part which is *not* strictly representative, simply because it is curious or interesting.

It is always well to make notes at the time of examination, as even the best photographs cannot show up the various characteristics as plainly as they appear to the eye.

Though it is useful for comparative results as far as possible always to keep a record at one fixed magnification (say,

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for instance, 50 diameters), it is better, in addition to this, to examine it at two or three different powers, at least with different eye pieces and frequently different objectives. An examination of the image on a clear-glass focussing screen with an eye piece will, however, give a still greater magnification.

PHOTOGRAPHING. *Focussing.* — This is by no means always easy, owing to the great dispersion of light in higher powers.

For locating the extent of the field, so as to find the position of any part of it, such as the edge of the piece, on an exact position on the plate; also, for giving a correct idea as to how the photograph will appear, a finely ground glass disc is very useful. With low powers, say up to 50 diameters, focussing can be clearly and sharply done on it.

To ensure very sharp focussing, a clear-glass screen and examination with a low-power eyepiece is preferable. Indeed, for very high powers this is practically indispensable.

We generally examine with both, especially since the latter, as already mentioned, gives a very good idea of the appearance under increased magnification.

The clear-glass is not indispensable, and has the disadvantage of being easily dulled by the breath, but it is more exact than merely holding the eyepiece in the right position.

We prefer, for comparative work, to always have the focussing screen at a fixed distance from the eyepiece, viz. 9 inches. This saves complication, especially in grain measurements.

Dry plates. — For cheapness and handiness, we use ¼ plates of a moderately rapid make.

Exposure. — From 35 to 110 diameters, 5 to 12 minutes with oblique light, and 4 to 9 minutes with the 45° reflector is usually sufficient; whilst for 300 diameters, 35 minutes with vertical light. Higher powers *pro rata.*

If prints are wanted very quickly, immediately the negative is rinsed it can be steeped in methylated spirit, and dried within 2 to 5 minutes. *After* taking the prints required, the usual washing can be done before storage.

Printing. — It has been considered best to be independent of daylight, and use bromide paper, a *slow* type giving den-

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sity being preferred. Enamelled paper gives better detail than matt surface.

The exposure varies from 2 to 5 seconds at 2 feet from an incandescent gas-light, but for weak or dense negatives can be humored to a large extent. With care, it is almost impossible to have a failure.

The print should be washed for at least one hour, though in urgent cases, where durability is of secondary consideration, less will suffice, and in this case, too, it can (like the negatives) be dried with the use of spirit.

Mounting. — "Slip-in" mounts and books will be found most convenient.

Average Time, in Urgent Cases.

	Minutes.		Minutes.
Sawing off section according to size of piece from which cut	1	to	5
Grinding and polishing	15	to	30
Polish examination	2	to	3
Etching and washing	3	to	5
Examination and photographing	7	to	10
Developing, fixing, rinsing, and drying negative	15	to	15
Printing	2	to	2
Developing, fixing, and rinsing print	15	to	20
Washing and drying in spirit	60	to	60
Total	120	to	150
	2 hours.		2½ hours.

Grain measurement and comparison. — For practical purposes, the exact measurement of the outline of all the grains with a planimeter, as described by Mr. Sauveur,* is too tedious.

The best general idea of the relative structure of the standard and the piece being examined is obtained by comparing the photographs of each *on exactly the same scale.* This is why uniform treatment of standards and samples at all points throughout has been advocated strongly, and if they are selected to correspond with each other within reasonable limits, the range of effects produced by uniform treat-

* *Transactions of the American Institute of Mining Engineers,* Vol. xxii, "The Microstructure of Steel."

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ment is not too great for reliance to be placed on the results of comparison.

For record it is desirable, as nearly as may be, to give a numerical value to the grain size, and this can be judged very readily and with a fair degree of accuracy by measuring the grains with scales drawn with Indian ink on clear glass, the surface of which has been prepared with retouching medium.

Micrometer scales are first photographed at the same magnification as the samples, the clear-glass scales being prepared from these.

Summing up and recording results, etc. — This should be carefully done as the examination proceeds, and will be found of great assistance in reproducing any given effect at a future time with precision, say, for instance, a duplicate print.

The records generally made, besides general remarks as to the appearance, opinion as to what the sample indicates, and anything else of note, are as follows:—

Number.
Etching medium.
Etching time.
Objective.
Eye-piece.
Magnification.
Illumination used.
Part of section photographed (whether outside or centre, etc.).
Exposure.
Print exposure.

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METALLOGRAPHIC NOTES.

Léon Gages on Microscopical Metallography. — Mr. Léon Gages concludes the second volume of his recent and remarkable treatise of the metallurgy of iron and steel by the following remarks of interest to metallographists: "The creators of the cellular theory* may to-day measure what has been accomplished since their first researches; they have laid down the foundations of a science which has rendered immense services to industry. Born but yesterday, microscopical metallography already distinguishes itself by an extraordinary fecundity; by analyzing the diathesis and diseases of metals, it will lead metallurgists and engineers to devise improved methods to avoid or to cure them."

S. Moutou on Microscopical Metallography. — In volume XXIII of *La Grande Encyclopédie*† recently published, under the heading of Metallurgy, four and a half pages are devoted to an illustrated description of the microscopical examination of metals. The article is signed by S. Moutou. It describes mostly the investigations of our contributors Osmond and Charpy.

Mr. Moutou begins his review with the following remarks: "Among the most useful and most interesting researches in the domain of metallurgy, which have been conducted in recent years, those of Osmond and Charpy should be especially noted. While chemical analysis indicates what are the elements which make up the composition of a metal, it deals only, as was so ingeniously said by Mr. Le Verrier, with the corpse of the metal, for the chemist subjects it to the action of reagents in which it is dissolved; he makes it pass through a series of combinations, and finally obtains either a

* Messrs. Osmond and Werth.

† Paris Société Anonyme de la Grande Encyclopédie.

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liquid or a minutely divided substance which no longer constitutes the metal to be studied. Chemical analysis tells us what is the composition of the metal, whether it is pure, whether the elements of which it is composed are present in the desired proportions, but it leaves us completely ignorant of its structure, of the grouping of the elementary components. Neither will it tell us anything concerning the conditions which may modify the structure, although the latter may play such an important part in metallurgy. To sum up, it throws no light upon what Mr. Le Verrier calls the anatomy and pathology of the metal. It is the microscope which will reveal to us its anatomy, and the pyrometer which will permit us to investigate its pathology."

The Crystalline Structure of Iron and Steel.* — Referring to the paper of Mr. Stead on the "Crystalline Structure of Iron and Steel," the following remarks of Mr. Osmond, taken from the *Journal of the Iron and Steel Institute*, 1898, Part I, are of interest. "He (Mr. Osmond) intended, in the course of a few months, to publish a work in which Mr. Stead's results would be considered and compared with those which he had collected on the same subject. Such an undertaking, however, required much time for its completion, and on this account he did not intend at present to discuss the paper. He wished, however, to cordially congratulate the author on the excellence of his work. The knowledge of the problem investigated, the choice of the means used, and the manipulative skill, together with the value of the facts brought to light, all combined to make the paper one of the most important of those which have been presented to the Institute. Metallurgy in its practical aspect ought to profit much by what the author had done, as it would enable some of the defective methods of annealing and working steel to be corrected. The value of microscopical research became more and more evident."

We shall all await with much interest and anticipation Mr. Osmond's coming work.

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* *The Metallographist*, Vol. I, page 289.

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The National Physical Laboratory. — It has been decided in England to maintain a "National Physical Laboratory" under the supervision of the "Royal Society." The government is to ask Parliament £12,000 for buildings and an annual grant of £4000. The present board includes two members from each of the following technical institutions: Institution of Civil Engineers, Institution of Mechanical Engineers, Institution of Electrical Engineers, Iron and Steel Institute, Society of Chemical Industry, and Institution of Naval Architects. The members from the Iron and Steel Institute are Sir W. Roberts-Austen and Sir Frederick Abel.

It is expected that metallographic investigations will form an important part of the work of this laboratory, and the publications of the results obtained will be awaited with great interest.

Metallography and the Iron and Steel Institute. — At the last meeting of the Iron and Steel Institute, held in Manchester, of the eleven papers that were read, five were based wholly or in part upon results obtained by metallographic methods. It is a forcible illustration of the advance of metallography and of its rapidly increasing importance as a factor of industrial value.

The papers referred to were:

1. "On the Constitution of Steel." By Professor E. D. Campbell, Ann Arbor, Mich.
2. "On Diffusion in Steel." By F. W. Harbord and Thomas Twynam, Cooper's Hill Engineering College, England.
3. "On Practical Microscopical Analysis for Use in the Steel Industries." By C. H. Ridsdale, Ginsbrough, England.
4. "On the Relation between the Structure of Steel and its Thermal and Mechanical Treatment." By Albert Sauveur, Boston, Mass.
5. "On the Present Position of the Solution Theory of Carburized Iron." By A. Stansfield, Royal School of Mines, England.

Article 4 was reproduced in the October 1899 issue of

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The Metallographist; article 1, 3 and 5 will be found in the present issue.

The Development of Metallography. — The revelations which the science of metallography has made in the domain of the physical structure of metals used in construction have been deemed so important that the German government has provided for a special investigator in this branch of applied science at the Charlottenburg Testing Laboratory. Professor Martens announces that Mr. E. Heyn, a former pupil of Professor Ledebur, has been engaged during the past year in a special line of investigations in metallography, or the microscopical study of etched metallic surfaces, the results of which are now given to the engineering world through the medium of the *Mittheilungen aus den Königlichen technischen Versuchsanstalten* in the form of a carefully written memoir, together with a large number of photogravure reproductions of photomicrographs.*

It is impossible to discuss the remarkable results which have been attained by Mr. Heyn without reference to the beautiful photomicrographs of etched sections which accompany his original memoir, but a general review of the examples submitted with the paper is most convincing evidence of the important relation which physical structure bears to strength and durability in metals of construction. It is quite possible for a material to yield an apparently satisfactory chemical analysis and yet be structurally very defective, while the crystalline arrangement must have a most important influence upon the questions of wear and endurance.

The whole subject of the strength and value of materials of construction is so intimately bound up with questions of a physical and chemical nature, that metallography forms a common ground upon which chemists, physicists and engineers must meet and exert united efforts, and nowhere is such work being done to better advantage than in the Royal Testing Laboratory at Charlottenburg.

The Engineering Magazine, June 1899.

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* Mr. Heyn's paper will be published in an early number of *The Metallographist.* — ED.

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Importance of Metallographic Methods. — The practical applications of these highly scientific researches are already being brought out by the study of the structure of steel rails, as compared with their behavior in actual service, and there is every evidence that the simple operation of polishing and etching properly selected sections, and a microscopical examination may hereafter be the powerful auxiliary of the chemical laboraty and the testing machine.

Engineering Magazine, April 1899.

Mr. H. SILVESTER, president of the Staffordshire Iron and Steel Institute, after reviewing in his inaugural address the various theories of the hardening of steel (a subject which has been exhaustively discussed in these columns) rightly says: "The history of the heat treatment of a piece of steel is embodied in its constitution, and these new methods of investigations are slowly but surely enabling the significance of the characters inscribed therein to be read aright."

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CORRESPONDENCE.

The Critical Ranges in Iron and Steel.

To the Editor of *The Metallographist.*

Sir:—I read with much interest Professor Howe's article on "the Critical Ranges in Iron and Steel" in the October 1899 number of *The Metallographist*, page 257.

It is very reasonable to suppose that the range A_2 has, strictly speaking, no inferior limit, that the ratio $\alpha:\beta$ tends at each temperature towards a certain limit, and that the interfering hysteretic resistances which prevent this limit to be reached are modified, in one way or the other, by cold working, by the percentage of manganese and by the magnetic field.

I had more especially in view, in publishing my paper entitled "What is the Inferior Limit of the Critical Point A_2?" in your July 1899 number, to connect our metallurgical experiments with those conducted by physicists, and I am pleased to see that I have already obtained a happy result. Professor Howe, who excels in drawing from a postulatum its logical inferences, and in suggesting their experimental verification, has quickly seen that the influence of the magnetic fields upon the critical points led us at once to a new subject of study which may be formulated as follows: are the mechanical properties of ferro-magnetic bodies modified, permanently or temporarily, in a magnetic field sufficiently powerful? Experiments in this direction are doubtless desirable. I myself had come to this conclusion.

Professor Howe suggests also some amendments to the proposition I formulated (*The Metallographist*, Vol. I, page 271) in order to indicate, by means of proper notations, the exact signification of the critical points. These amendments appear to me justified and well conceived; they certainly improve my original propositions, and will facilitate the exposition and the discussion of the delicate problems with which we are dealing.

F. OSMOND.

PARIS, December 5, 1899.

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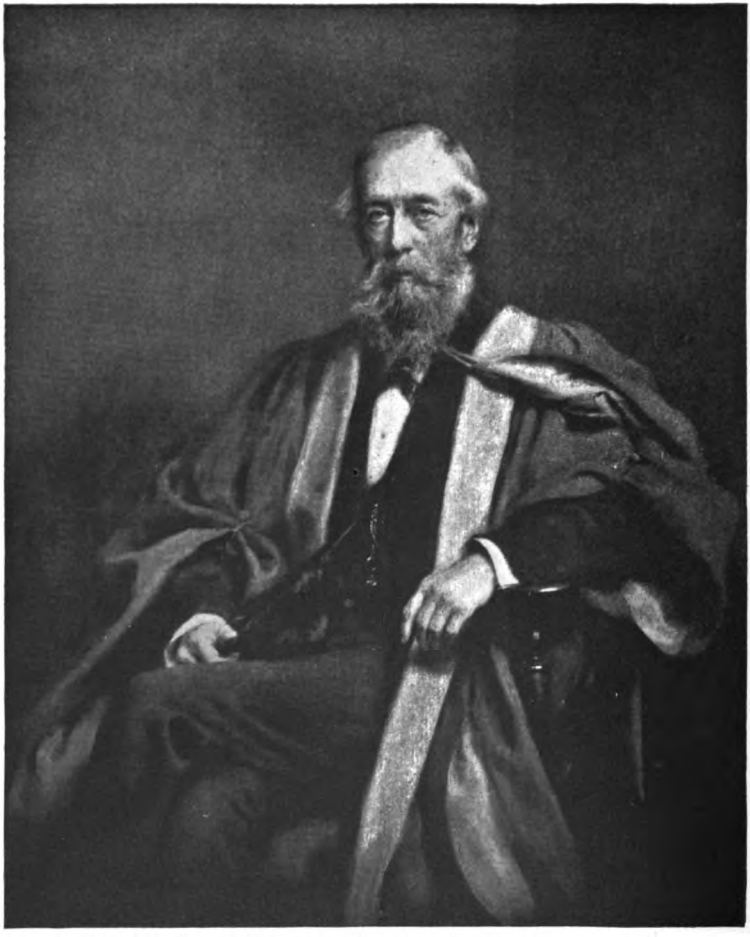

HENRY CLIFTON SORBY.

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The Metallographist.

A QUARTERLY PUBLICATION DEVOTED TO THE STUDY OF METALS, WITH SPECIAL REFERENCE TO THEIR PHYSICS AND MICROSTRUCTURE, THEIR INDUSTRIAL TREATMENT AND APPLICATIONS.

Vol. III. APRIL 1900. No. 2.

HENRY CLIFTON SORBY.*

HENRY CLIFTON SORBY was born at Woodhouse, near Sheffield, England, May 10, 1826. He was educated at the Sheffield Collegiate School and by private tutors.

Concerning his early education Dr. Sorby himself says: "From my early boyhood I had a very great taste for different departments of Science. I went to the Collegiate School and obtained there a prize for arithmetic. That prize was entitled 'Readings in Science.' I took a very great interest in that book, and it had a very large share in causing me to direct my attention to scientific research. Moreover after leaving the Collegiate School I had a mathematical tutor, the Rev. Walter Mitchell, who had been brought up a medical student. He was a fairly good anatomist and also a fairly good chemist; and we used to work together, not only at mathematics, but at anatomy and chemistry. That also greatly influenced my subsequent proceedings. When he left, I had pretty well made up my mind that I would devote myself to a scientific career, and I worked at mathematics, optics, chemistry, anatomy and water-color drawing. My early training in all these things has turned out to be of the greatest possible value in what I have since done. I may

* For the information required to compile this biography, we are indebted to the great kindness of Mr. R. A. Hadfield.

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say that I worked not to pass an examination, but to qualify myself for a career of original investigation."

In 1846 Dr. Sorby lost his father and in 1853 he removed with his mother to Broomfield, near Sheffield, where he still resides. In 1874 his mother died, and since 1878 he has spent half of each year on board his yacht the "Glimpse."

The following partial list of the honorary positions occupied by Dr. Sorby and of the numerous distinctions awarded to him, testify of the high esteem in which he is held, not only in his own country, but also in that land which knows no geographical boundary, "the scientific World." The number and character of these distinctions constitute the best possible evidence of the recognition, by his contemporaries, of the invaluable services rendered by Dr. Sorby in the various departments of Science in which he directed his researches.

In 1852 he became president of the Sheffield Literary and Philosophical Society, in 1853 fellow of the Geological Society, in 1857 fellow of the Royal Society. In. 1869 he was awarded the Woollaston medal by the Geological Society of London. In 1872 he was the first to receive the Boerhaave medal, given once in twenty years by the Dutch Society of Science, to him who has done most to advance geology and mineralogy in the previous twenty years. The Royal Society awarded to him, in 1874, one of the two large gold medals given annually by Her Majesty the Queen.

From 1875 to 1877, Dr. Sorby was president of the Royal Microscopical Society. In 1877 he was chosen as the first president of the then newly formed, Mineralogical Society of Great Britain and Ireland. In 1878 and 1879 he was president of the Geological Society.

In 1879 the University of Cambridge conferred on him the honorary degree of LL.D.

Dr. Sorby is a foreign member of the Mineralogical Society of St. Petersburg, one of the 18 foreign members of the "Accademia dei Lincei" of Rome, the oldest scientific society in the world, and of many other societies.

In his own town, he is president of the council of Firth College, chairman of the Technical School, president of the School of Arts, of the Mechanics Institute, etc.

Dr. Sorby's writings are characterized by great clearness,

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precision, thoroughness and originality, and bear the mark of work done with remarkable persistence and self-reliance. He had to maintain some of the revolutionary new theories which he advanced, in the face of much opposition on the part of men of science, many years his seniors, and in exalted scientific position. In every instance, however, he came out victorious, and his theories are now taught as accepted scientific facts and have been most fruitful in important results.

Dr. Sorby wrote his first paper in 1847 and since then has contributed some 150 papers to various scientific societies and publications, a striking evidence of the remarkable activity and fertility of the investigator.

It may be said that his chief studies were connected with the application of the miscroscope to every department of natural science in which that instrument now plays an important part, geology and mineralogy being the subject of his special attention. He devoted much time to the study of the microscopical structure of crystals, rocks and minerals, being the first one to prepare thin sections of rocks, suitable for examination by transmitted light. He was the first one to devise a spectrum microscope and may be said to be the creator of micro-spectroscopy. He studied the coloring matter of animals and plants with valuable results. Dr. Sorby was the first one to demonstrate that slaty cleavage is due to mechanical pressure, a fact of very great geological importance.

Finally, and here we come to a subject of more direct interest to the readers of this journal, Dr. Sorby gave his attention to the microscopical structure of iron and steel. He tells us in the following interesting remarks how he was led to take up this study: "It was a natural thing that I should be led from the study of the microscopical structure of rocks to that of meteorites, and in order to explain the structure of meteoric iron I commenced the study of artificial irons. I began this work in 1863, and in 1864 gave an account of it at the British Association. This attracted no attention for 22 years, but in 1887 the Iron and Steel Institute requested me to again take the matter up, and appointed Dr. Percy, Sir Henry Bessemer, and myself to decide what was the best way of illustrating a complete paper on the subject. That was done, and since then the microscopical investigation of iron and steel has been largely recognized as a

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most important means of studying them. In those early days, if a railway accident had occurred and I had suggested that the company should take up a rail and have it examined with the microscope, I should have been looked upon as a fit man to send to an asylum. But that is what is now being done; and very gratified am I to think that our member, Mr. Thos. Andrews, is doing most admirable work in connection with this subject. What I really proved was that various kinds of iron and steel are varying mixtures of well-defined substances, and that their structure is in many respects analogous to that of igneous rock. I also took specimens of iron and steel and acted upon them with acid so that it was possible to print from them as from types, and show many interesting points connected with their structure."

It is seen then that it is almost accidentally that Dr. Sorby undertook the study of the microstructure of iron and steel, and that the work which he did in this direction constitutes a very small part of his total achievements.

This work, however, in a hitherto wholly unexplored field, was conducted with all the skill, thoroughness and clear-mindedness of this accomplished scientist. The reader knows how it was then taken up by other investigators and how productive of important results it has been.

The complete list of Dr. Sorby's writings dealing with the microstructure of iron and steel is given below in chronological order:

On a New Method of Illustrating the Structure of Various Kinds of Steel by Nature Printing. — Sheffield Literary and Philosophical Society, February, 1864.

On the Microscopical Photographs of Various Kinds of Iron and Steel. — *British Association Report*, 1864, Part 2, page 189.

On the Microscopical Structure of Meteorites and Meteoric Iron. — *Proceedings of the Royal Society*, Vol. XIII, page 333, and *British Association Report*, 1865, Part 1, page 139.

On the Microscopical Structure of Iron and Steel. — In Dr. Lionel Beale's *How to Work with a Microscope*, 4th edition, 1868, pages 181–183.

The Microscopical Structure of Iron and Steel. — Lecture delivered at the Firth College, Sheffield, October 20, 1882. *The Engineer*, Vol. LIV, page 308, October 27, 1882.

On the Application of Very High Powers to the Study of Microscopical Structures of Steel. — *Journal of Iron and Steel Institute*, Vol. I, 1886, pages 140–144.

On the Microscopical Structure of Iron and Steel. Seventeen illus-

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trations.—*Journal of Iron and Steel Institute*, Vol. I, 1887, pages 255–288.

Microscopical Analysis of Iron and Steel.—Lecture before the Sheffield Society of Engineers. Abstd. in *Engineering*, Vol. LI, page 73, January 16, 1891.

Describes the Results of His Investigations on the Microscopic Structure of Iron and Steel.—Lecture before the Derby Society of Engineers, delivered October 28, 1892, through *Industries*, Vol. XIII, pages 466–467. Abstd. in *Journal of Iron and Steel Institute*, Vol. I, 1893, pages 365–366.

For fourteen years after the publication of his first paper no other scientist seems to have deemed the subject of sufficient importance to warrant investigation, for there is no record of any work having been done by others until 1878, when Prof. Martens of Berlin, published the first of a series of valuable contributions to the microscopy of iron and steel.

The two masterly papers which Dr. Sorby read before the Iron and Steel Institute, however, in 1886 and 1887, although deemed of little practical value, at the meetings, soon aroused the interest of many investigators, and were the signal for great activity in the field he had so brilliantly started to explore.

In these two papers Dr. Sorby describes at length the structure of iron and of various grades of steel as revealed by the microscope, illustrating his remarks with some photomicrographs which are all the more beautiful when it is considered how little had then been done in photographing opaque objects. He also describes his method of preparing the sections for examination. It is interesting to recall that it was not until 1885 that Dr. Sorby discovered the true nature of the constituent which he termed the "pearly constituent," and which we now call "pearlyte," because not until then did he use high power in his inquiry. The remarkable features of this constituent, as seen under high magnification, could not fail to arouse intense interest and surprise in so consummate a microscopist, for it was not then known that these characteristics of the structure of pearlyte are those of all eutectic alloys and indeed of cryohydrates, those eutectic mixtures of aqueous saline solutions.

In concluding this brief outline of Dr. Sorby's achievements, a few remarks of the editor on some previous occasion may perhaps be quoted:

"So great was the caution and so keen the observing power

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of this eminent microscopist that the foundation he laid for the new structure, although thoroughly and critically tested, has been found to be of a most trustworthy character. Further researches undertaken since by many investigators have confirmed, in their essential features, the truth of his observations and deductions concerning the microscopic constituents of iron and steel; and although the methods for the preparation of the samples have been somewhat modified and improved, still in their vital parts they remain such as he devised them. Whatever has been accomplished since in microscopic metallography has been done by following his footsteps. To Dr. Sorby and to him alone is due the pioneer's honor."

THE CRYSTALLINE STRUCTURE OF METALS.*

By J. A. EWING and WALTER ROSENHAIN.

THE microscopic study of metals was initiated by Sorby,† and has been pursued by Arnold, Andrews, Behrens, Charpy, Howe, Martens, Osmond, Roberts-Austen, Sauveur, Stead, Tschernoff, Wedding, Werth and others.‡ The work of these authors has demonstrated the value of the microscope in metallurgy, not only as an aid to analysis, but as means of observing structure. The structure of pure metals, of metals containing small quantities of foreign matter, and of alloys, has been made the subject of microscopic examination, and important conclusions have been reached. The work to be described in this paper proceeds on the same general lines. A large part of it deals with a branch of the subject which has not hitherto received much notice, namely, the effects of strain. The writers believe that they have established the fact that the structure of metals is crystalline even under conditions which might be supposed to destroy crystalline structure. They have found that the plastic yielding of metals when severely strained occurs in such a manner that the crystalline structure is preserved. The observations to be described show how crystalline aggregates ex-

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* Bakerian Lecture, May 18, 1899. *Philosophical Transactions, Royal Society of London*, Vol. CXCIII, page 353.

† *Roy. Soc. Proc.*, Vol. XIII, page 333.

‡ For the bibliography see *The Metallographist*, Vol. I (1898), page 168.

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hibit plasticity, and how, after straining, a metal continues to be a crystalline aggregate. The distinction which is often drawn between crystalline and non-crystalline states in metals appears to be unfounded.

Except for a few simple innovations, the methods of experiment used in this research, especially as regards the preparation of specimens, do not differ materially from those of earlier workers. The specimens were first polished on commercial emery-paper which had been previously rubbed on a piece of hard steel in order to remove the coarser particles. They were finished on a rapidly revolving disc coated with fine wash-leather and charged with a thin paste of rouge and water. For most purposes the finest jeweller's rouge is suitable, but in special cases

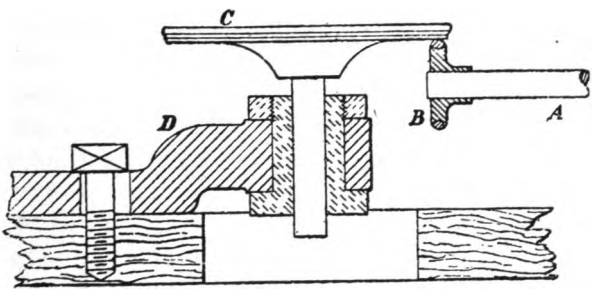

Fig. 1.

we resorted to the use of peroxide of iron obtained by precipitation from a solution of pure ferric chloride.

The polishing machine is shown in diagram-section in Fig. 1. *A* is the spindle of an electro-motor carrying a small driving disc *B*, fitted with a rubber ring to increase the driving friction. The polishing disc *C* is horizontal and has a vertical axis running in a bearing in the casting *D*. The under side of the polishing disc bears upon the driving wheel *B* and takes motion from it. When it is desired to stop temporarily the polishing disc is raised out of contact with the driving wheel. The casting *D* is held to the base-board by a bolt passing through a slot which allows the disc to be brought nearer to the motor, so that it may bear upon the driving wheel at any desired distance from its axis, thus giving a means of varying the speed. More usually, however, the speed was varied by regulating the current in the motor.

With metals which are very liable to tarnish, wet polishing

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has to be avoided; in some cases dry rouge, and in others rouge moistened with a little paraffin, may be used — the latter we found particularly useful in polishing copper. But in some metals, especially the more fusible ones, such as lead, zinc, and tin, it is difficult to obtain a satisfactory surface by any method of polishing. In most of these cases, however, a method of obtaining a good surface entirely without polishing becomes available. This consists in pouring the molten metal on a smooth body, such as glass or polished steel, in contact with which it is allowed to solidify. We have, as a rule, used glass for this purpose, and in spite of frequent failures, due to fracture of the glass or to less obvious causes, we find that it is generally practicable to get a good specimen in this way with much less trouble than is required to produce a specimen of iron or steel by the ordinary process of polishing. This method of "glass casting" cannot well be applied to metals which oxidize at temperatures below their melting points, and for metals which have a very high melting point a smooth body other than glass must be used. The metals most readily treated by casting against a smooth surface are gold, bismuth, cadmium, lead, tin, zinc, and fusible alloys.

In the examination of lead another method of obtaining a good surface, without either melting or polishing, was also used. A face of the specimen was freshly cut to remove the tarnish, and was then pressed against a smooth surface of plate-glass. Whenever a sufficient pressure could be reached without breaking the glass a very beautiful surface was obtained. In some cases polished steel was used as the smooth object against which the metal was pressed.

The polished surfaces were, in general, slightly etched before microscopic examination, in most instances by dilute nitric acid. Frequently, however, no etching was resorted to, especially in observations in dealing with the effects of strain. It has been pointed out by Charpy that straining, by the relative displacement which it brings about among the crystalline grains, serves to reveal the structure. Our observations confirm this, and in some cases we found that a better investigation of the effects of strain could be made when the surface was not etched.

Most of the microscopic observations were made with Zeiss' apochromatic lenses, a 2-mm. homogeneous immersion lens of 1.40 aperture being employed for high-power work, with compen-

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sating eye-pieces magnifying from 4 to 18 times, giving in direct vision a total magnification up to 3000 diameters. "Vertical" illumination was generally used, the objective serving as condenser, but in some cases the specimens were examined under oblique light. Photographic records of the most interesting features were obtained, some of which are reproduced in this paper. The source of light was an arc lamp, the beam from which was condensed on the illuminator through a "Gifford" screen which allowed only a very limited portion of the spectrum to pass. Most of the high-power photographs were taken with a magnification of 1000 diameters; in a few instances it was 4000 diameters or more.

It is well known that when the polished surface of a metal, such as gold or iron, is lightly etched, and is then examined by means of normally reflected ("vertical") light, the surface appears divided up into a number of areas separated by more or less polygonal boundaries. These areas are the sections of the crystalline grains which constitute the mass of the metal; the boundaries between them have been made evident by the differential action of the acid which has produced differences of level by attacking one grain more energetically than its neighbor. Fig. 2 illustrates this appearance in ordinary iron. There the black patches are due to the presence of slag, and the black lines forming the boundaries are due to differences of level between the grains. Each of the short sloping surfaces which connects one grain with another appears black because it does not reflect the normally incident light back into the tube.

It is also well known that a further differential action of the acid is to reveal a difference of texture between the grains. This is visible in Fig 2, but is much more pronounced when the surface is examined under oblique light. When the light is uni-directional or nearly so, the various grains differ very much in brightness and color — some are almost black, while others shine out brightly; if, however, the incidence of the light or the orientation of the specimen be changed, other grains shine out strongly, while those previously bright become dark. This effect was first observed in gold by Arnold (*Engineering*, February 7, 1896), who accounted for it by considering that each crystalline grain is built up of a very large number of what he (following Andrews) calls secondary crystals, which have different orientations in different

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by Osmond and Roberts-Austen (*Phil. Trans.*, A, Vol. 187, pages 424-5), who speak of little crystals, the general orientation of which remains constant in the area of each grain." Many beautiful illustrations of the same effect in iron have been given by Stead, along with a clear discussion of the cause to which this appearance is to be ascribed (*Journal of the Iron and Steel Institute*, 1898). A striking instance is shown in his photographs of steel containing about 4½ per cent of silicon. The fractured ingot of this material exhibits large crystals, and by deeply etch-

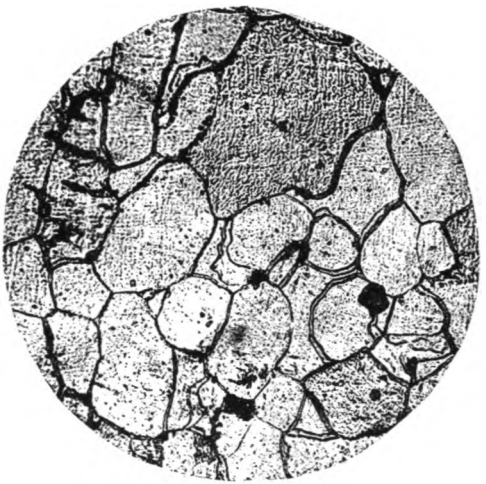

Fig. 2. Nearly pure commercial iron (transformer plate). Polished and etched. Magnified 200 diameters. Vertical light.

ing a polished surface he has obtained a fine development of the regularly oriented elements of which the crystalline grains are built up, on a scale so large as to be clear with little or no magnification.

These observations have made it plain that each of the grains which appear on the polished and etched surface of a metal is simply a crystal, the growth of which has been arrested by its meeting with neighboring grains. The irregular boundaries of the grains are determined by these meetings. We may imagine the grains to grow from as many centres or nuclei as there are but here and there the acid has etched out minute pits showing grains. Substantially the same view is expressed in other terms

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grains. Each grain is built up of similarly oriented parts, but the orientation changes from grain to grain. Etching a polished surface develops a multitude of facets which have the same orientation over the surface of any one grain, but different orientations in different grains. Seen under oblique illumination these facets show themselves to be similarly oriented in each grain by the uniform manner in which the grain reflects light, and by the disappearance of brightness over the whole surface of the grain when the incidence of the light is changed. The mass of the grain consists of similarly oriented elements; as to the size of the elements no assumption need be made. The facets which are developed by etching do not, in general, appear of constant size; it is to the constancy of their orientation that the effect is due.

A striking illustration of how a metal crystallises by the simultaneous building up of groups of elements from a number of centres, the elements in each group being similarly oriented, while the orientation varies from group to group, may be seen in a cake of solidifying bismuth from which the still molten metal has been poured away. The operation then goes on upon a scale so large that no magnification is required to make it apparent.

The references given above will show that there is nothing novel in this view of the structure of metals. Several of the authors' observation may, however, be mentioned as affording additional evidence that the structure of metals in general consists of crystalline grains built up in the manner which has been described. One class of evidence has been obtained by an examination of specimens of iron which had been deeply etched under very high magnification, i.e. 2000 to 3000 diameters. Under favorable circumstances it is possible to resolve the minute structure to which the peculiar reflection of oblique light is due. The face of each grain is then seen to be covered with minute projections resembling scales, more or less square or oblong in shape and similar and similarly oriented over the entire face of one grain. Fig. 3 is a photograph of this appearance as seen in nearly pure wrought iron: it may be compared (as Mr. Stead compares a like appearance occurring on a large scale in silicon steel) to the arrangement of slates on the roof of a house. In other cases the action of the acid is different; the general surface of a grain cannot be resolved under the highest powers,

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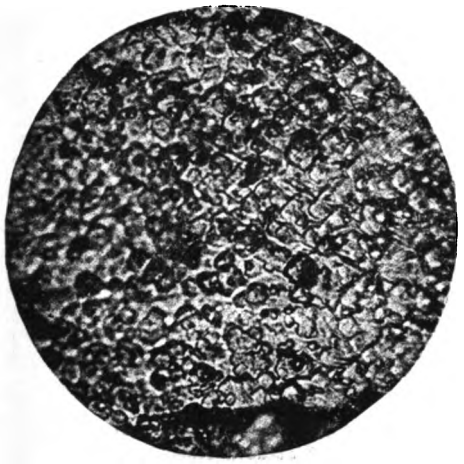

Fig. 3. Crystalline grains of Iron after etching.
Magnified 1500 diameters. Vertical light.

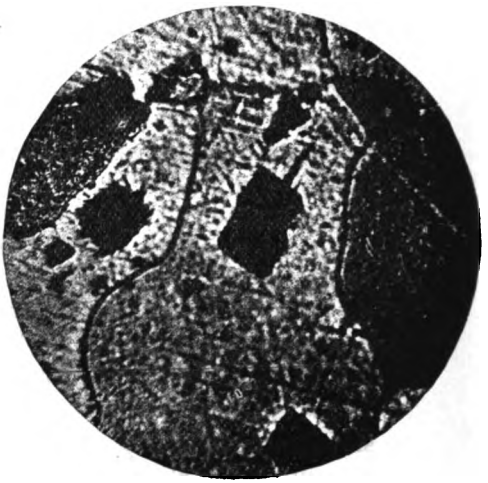

Fig. 4. Etched pits in Swedish Iron.
Magnified 1000 diameters.
Vertical light.

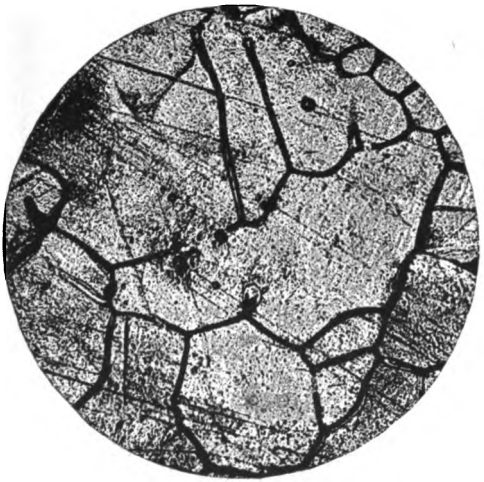

Fig. 5. Glass-cast surface of Cadmium showing crystalline boundaries and air-channels.
Magnified 100 diameters. Vertical light.

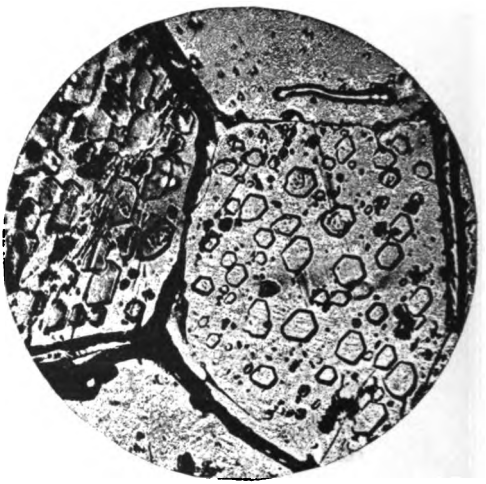

Fig. 6. Air-pits on a glass-cast surface of Cadmium.
Magnified 1000 diameters. Vertical light.

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a distinct geometric form. All these pits found over the face of one grain are similar and similarly situated figures, but the shape orientation of the pits changes as soon as a boundary is crossed. This is shown in a very striking way where a comparatively large pit crosses the boundary so that a portion appears on each side. Each portion preserves its proper shape and orientation, and there is consequently a marked angle in the sides of the pit where the sides cross the boundary. The shape of all these pits in iron is consistent with the assumption that they are plane sections of cubes or octahedra.

Fig. 4 is a photograph of such etched pits in Swedish iron.

A good development of geometrically etched pits is not very readily obtained: in some specimens of iron they occur with much greater readiness than in others, and this occurrence is to some extent an accident of etching. Possibly the presence of a minute quantity of impurity in the iron is an essential factor, but we have no evidence on the subject. Geometrical etched pits are a well known phenomenon in non-metallic mineral crystals. Baumhauer* finds that they have a definite relation to the crystallographic nature of the crystal upon which they occur — but the facets developed by etching often lie in planes which are not parallel to the natural faces of the crystal. He finds that these etched pits, though generally truly geometrical, frequently show curved or irregular outlines which he attributes to local concentration of the acid. We find that curved or irregular outlines often occur in the larger etched pits in iron, and in view of Baumhauer's observations it is clear that they cannot be taken to affect the evidence for the strictly crystalline nature of the metal, since similar appearances are to be found in bodies that are characteristically crystalline.

When metals are cast against glass or other smooth bodies, to get a surface fit for microscopic examination, evidences of crystalline structure appear apart from anything that is shown by etching. The cast surface generally shows clearly, without any etching, the boundaries between the crystalline grains. Good examples of this are found in cadmium, lead, tin, and zinc. In some instances the boundaries between the grains are emphasized in a remarkable way by the accumulation there of air or of gas

* Baumhauer, *Resultate der Aetzmethode in der Krystallographischen Forschung*. Wilhelm Engelmann, Leipzig, 1894.

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given off by the metal during solidification. The boundaries then appear as more or less deep and wide channels. As the growth of the crystalline grains proceeds, most of the air or gas which has been entrapped between the glass and the metal, and most of the gas given out from solution in the metal itself, tends to accumulate at the boundaries, which are the last parts of the surface to undergo solidification. A network of channels consequently appears on the surface next the glass; in general these channels coincide with boundaries, but occasionally a channel forms a *cul-de-sac* or terminates in a large cavity which is obviously a bubble or blow-hole. Fig. 5 shows the appearance of a surface of cadmium cast against glass under conditions of temperature favorable to the formation of these channels. By etching or straining the specimen it is easy to prove that each channel (except when it is a *cul-de-sac* formed by excess of gas) coincides in general with a real boundary between the crystalline grains. The true boundary is merely the trace of a surface on the plane of casting, but it may be broadened out in this way, by the presence of gas, into a shallow channel of considerable width. It is only in certain conditions of temperature, on the part of the metal and of the body against which it is cast, that any marked development of such channels is obtained. Under most conditions, however, it is easy to distinguish the intergranular boundaries in the cast surface, either by the presence of some gas there or by slight differences of level between one grain and the next.

Occasionally the faces of some of the grains in the surface cast against glass are covered with a number of very minute pits, whose true character appears only under a magnification of about 1000 diameters. They are then seen to be pits of definite geometrical form both as to outline on the surface and as to inner facets. Figs. 6, 7 and 8 are photographs of these pits in glass-cast cadmium. It will be seen that they are systems of geometrical figures which remain similar and similarly situated over the entire surface of a single grain, but change in character from one grain to the next. These pits appear to be excessively small bubbles or blow-holes which have taken a geometrical shape, forming, in fact, negative crystals. During the crystallisation the cristalline elements have built themselves around the gas bubbles in regular orientation, finally leaving the pits as we see them. In support of this view the appearance seen on Fig. 7 may

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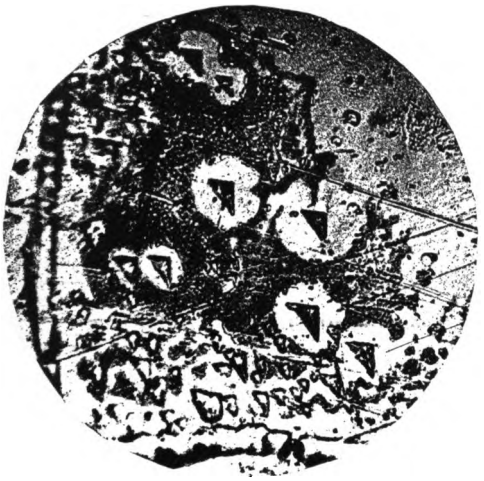

Fig. 7. Air-pits on a glass-cast surface of Cadmium.
Magnified 1000 diameters. Vertical light.

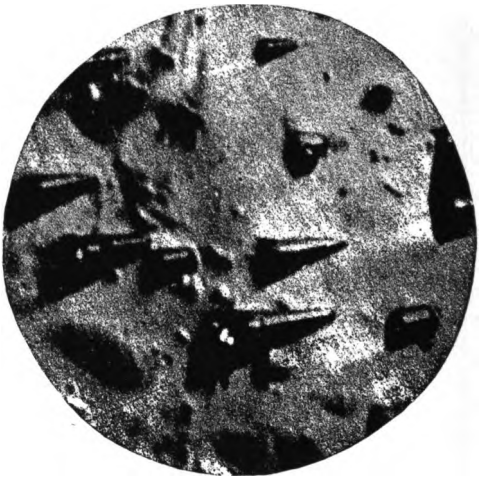

Fig. 8. Similar pits to those in Figs. 6 and 7.
Magnified 4200 diameters.
Vertical light.

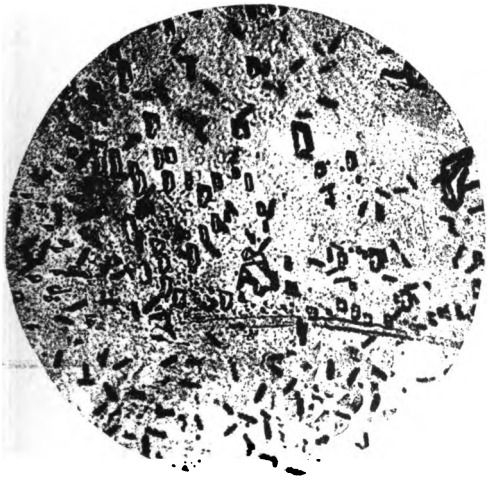

Fig. 9. Air-pits in glass-cast Tin.
The irregularly oriented black patches are crystals of an impurity.
Magnified 1000 diameters. Vertical light.

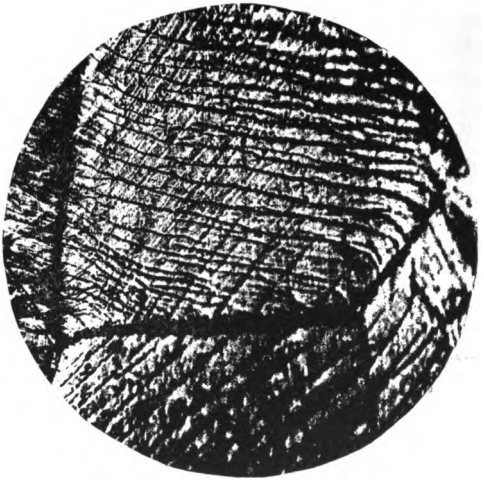

Fig. 11. Slip-bands in Swedish Iron strained by tension.
Magnified 400 diameters. Vertical light.

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be cited; the pits are seen to be surrounded by a halo or circular patch of bright surface — due apparently to the absorption by these larger geometrical bubbles of the numerous tiny bubbles that dot the surface elsewhere.

The photographs show these "air-pits" in cadmium cast against glass under a magnification in most cases of 1000 diameters, and in one case (Fig. 8) of 4200 diameters. The characteristic of the pits is that they are similar and similarly oriented over any one grain, but on passing from one grain to another, across a boundary, the orientation of the pit is found to have changed. Good examples of this are seen in Fig. 6, where the boundaries are widened out into channels through the presence there of air or gas in the manner described above. This characteristic of the air-pits is, of course, in complete conformity with the view already stated of the crystalline structure of metals. An additional photograph of air-pits in cadmium is shown in another connection in Fig. 23.

Examination of the air-pits in cadmium shows that the forms may be accounted for as sections of hexagonal prisms. It may be concluded that each crystalline element of which the grains of cadmium are built up is a hexagonal prism with plane base.*

These air-pits are not very readily developed, and the precise conditions which determine their appearance are not easily specified. Many specimens of the metal may be cast without obtaining them. That they are not, however, peculiar to cadmium is certain, for we have found them also in tin and in zinc. Fig. 9 shows them in a glass-cut surface of tin.

The same photograph illustrates another interesting feature. The surface of the tin is seen to be covered with a multitude of small dark crystals irregularly disposed. These are evidently inclusions of some foreign matter, which we conjecture to have been sulphide, because it was observed that they appeared in large numbers after the metal had, during melting, been directly exposed to a gas flame. The foreign crystals have no definite orientation, and are quite independent of the orientation of the metal within

* It may be added in this connection that Behrens remarks on the frequency of six-sided forms in the polygonal boundaries of the crystalline grains of cadmium. The form of boundary is, however, of little service in determining the character of the crystallisations within the grain. (See his work *Das Microscopische Gefüge der Metalle und Legierungen*, Leipzig, 1894.)

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the grains in which they are embedded. The crystallisation of the metal has proceeded around them without check or disturbance, just as in iron containing slag the growth of each crystalline grain ignores the presence of that impurity. It is well known that a slag band is often seen running right through a crystalline grain without affecting the uniformity of orientation of the elements of which the grain is built up.

Although is must be admitted that a really good development of geometrical bubbles such as those shown in the photographs is exceptional and cannot as yet be reproduced at will, the authors have observed that nearly all bubbles or blow-holes found on surfaces prepared in this way show a distinct tendency to geometrical shapes. A truly round bubble is rarely or never found, and even the larger bubbles often show a multitude of distinct facets reflecting light at different angles.

The occurrence of such geometrical pits in surfaces of metals that have never been polished or etched may be taken as very strong evidence in support of the view that the crystalline grains of metals are built up of crystalline elements which are similarly oriented throughout the mass of each grain.

The experiments now to be described were intended to throw light on the nature of plastic strain in metals, and the results obtained are interpreted by the aid of the theory of the crystalline structure of metals, of which an outline has been stated at the beginning of this paper. Their complete agreement with that theory affords further confirmation of its truth.

It has long been known that when a specimen of iron or steel with a bright smooth surface is strained sufficiently to give it permanent set the smoothness of the surface is destroyed. A microscopic examination of the surface shows, as Charpy has pointed out, that the crystalline grains become visibly differentiated from one another by straining, and the effect is in this respect not unlike that which etching would have produced. There is, however, a further effect of straining, which will be described immediately.

It is also well known that when a piece of severely-strained metal is polished and etched, as for instance a bar which has had its section reduced by hammering or rolling in the cold state, the crystalline grains are found to have changed their form. They are lengthened in the direction in which the piece was extended

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and shortened in the direction in which the section has contracted. Accordingly, a severely-strained piece is readily recognized on polishing and etching it, on account of the shape of its crystalline grains. In the strained specimen these are found to have a direction of predominating length according to the direction of strain, while in the unstrained specimen there is no direction of predominating length. Further, it is well known that in a strained specimen elongated grains are not found after the strained state has been relieved by annealing. The effect of rolling or hammering is often spoken of as a conversion of the metal from a crystalline to a "fibrous" state. In the present writers' view this language is misleading, and the physical conception underlying it is a mistaken one.

In the first experiments on the effects of strain we aimed at keeping a particular place on the polished and etched surface of the specimen under continuous observation while the specimen was being strained. This was accomplished by constructing a small straining machine which could be attached to the stage of the microscope, and by which strips of sheet metal could be gradually extended until they broke. Fig. 10 shows the arrangement which was used. The stress was applied by a screw which could be turned by hand while the specimen was under observation, and any displacement of the particular grains on which the microscope was focussed could be made good by means of the traversing screws in the mechanical stage. With this apparatus it was easy to keep the same group of crystalline grains under observation from the first application of stress until the specimen was broken, and to obtain photographs of the same group at any number of stages during the strain. The first specimens observed in this way were strips of sheet iron, of the nearly pure kind supplied for use in electrical transformers. The following account of what we have observed may be read as applying not only to iron but to other metals. Within the limit of elasticity no effects of strain are detected, but when the yield-point is reached a remarkable change is seen on the surface of the crystalline grains. As soon as plastic deformation begins the faces of the grains show fine black lines, and as the strain increases these lines increase in number; they are more or less straight and parallel in each grain, but are differently directed in different grains. The first lines to appear are those approximately trans-

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verse to the pull, but as the strain increases systems of inclined lines appear on other grains. With further straining some of the grains begin to show more than one system of such lines, and eventually two, three, and even four systems of intersecting lines on a single grain may be seen.

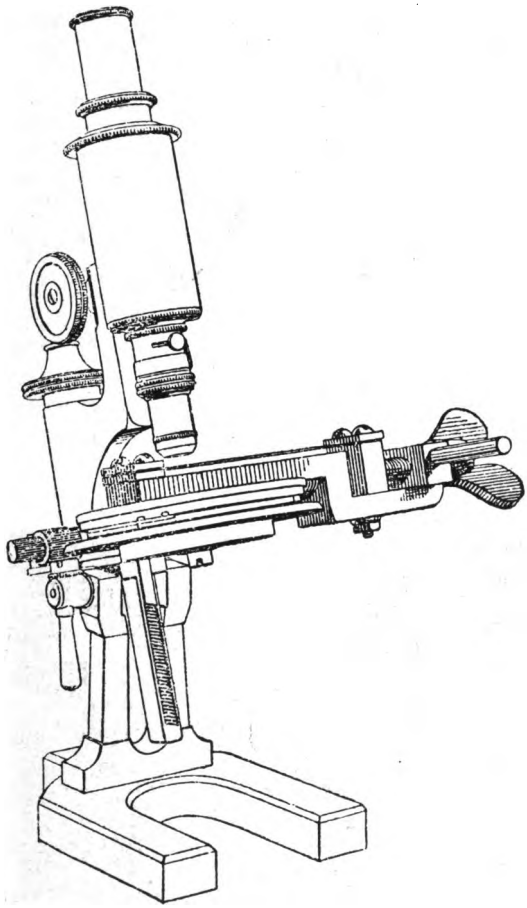

Fig. 10.

A characteristic example of these lines as exhibited by iron is shown in Fig. 11, which is a photograph of a piece of Swedish iron strained by pull. Figs. 12 and 13 are two views of the same group of crystalline grains in another piece of Swedish iron, Fig. 12 being taken before straining, and Fig. 13 after a con-

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Fig. 12. Fig. 13.
Same crystalline grains in Iron before and after straining.
Magnified 200 diameters. Vertical light.

Fig. 15. Strained Swedish Iron.
Magnified 300 diameters. Vertical light.

Fig. 16. Same field as in Fig. 15,
Seen under oblique light.

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siderable amount of straining. When the piece is much strained the surface becomes so rough as to make it difficult to secure a satisfactory photograph.

The appearance of the grains after straining so closely resembles that of a crevassed glacier that the black lines might be taken for cracks. But from the outset it was clear that they could not be actual fissures. A piece of strained iron, when it has been allowed to rest for a time, or has been heated to 100° C.,* recovers its original elasticity and more than its original strength, yet the dark lines do not disappear under such treatment. Further, if a specimen showing these lines be re-

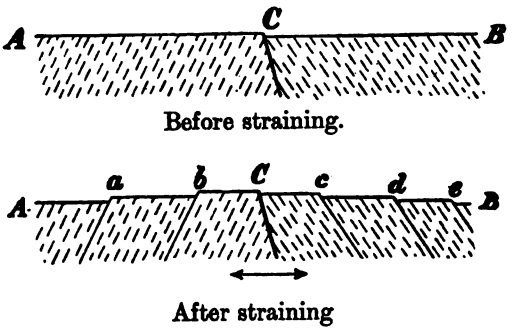

Fig. 14.

polished the lines disappear, and even light etching does not reproduce lines of the same nature.

The real character of the lines is apparent when the crystalline constitution of each grain is considered. They are not cracks but steps in the surface. These steps are due to slips along the cleavage or gliding planes of the crystals.†

The diagram, Fig. 14, is intended to represent a section through the upper part of two contiguous surface grains, having cleavage or gliding planes as indicated by the dotted lines, *AB* being a portion of the polished surface, *C* being the junction between the two grains.

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* See J. Muir, "On the Recovery of Iron from Overstrain," *Phil. Trans.*, A, 1899.

† See *Roy. Soc. Proc.*, March 16, 1899 (Vol. LXV, page 85), where the authors have published a preliminary account of some of these observations.

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When the metal is strained beyond its elastic limit, as say by a pull in the direction of the arrows, yielding takes place by finite amounts of slips at a limited number of places, in the manner shown at *a, b, c, d, e.* This exposes short portions of inclined cleavage or gliding surfaces, and when viewed in the microscope under normally incident light these surfaces appear black because they return no light to the microscope. They consequently show as dark lines or narrow bands extending over the polished surface in directions which depend on the intersection of the polished surface with the surfaces of slip.

The correctness of this view is demonstrated when these bands are examined under oblique light. When the light is incident at only a small angle to the polished surface, that surface appears for the most part dark; but here and there a system of the parallel bands shines out brilliantly in consequence of the short cleavage surfaces which constitute the bands having the proper inclination for reflecting light into the microscope. The groups of bright bands which are seen under oblique light are readily observed under the microscope to be exactly coincident with the black bands seen under vertical illumination. Figs. 15 and 16 are photographs of the same strained crystalline grains of iron under vertical light and under oblique light.

Rotation of the stage upon which the strained specimen is fixed makes the bands on one or another of the grains flash out successively with kaleidoscopic effect. When the specimen is rotated through 180° from the position in which the lines show brightly on one particular grain, the lines on that grain do not shine out again, though they may be visible as black lines on a very faintly luminous background; this is important as proving that the lines are not due to either furrows or ridges, but to steps in the surface. In this respect there is a striking contrast between the slip-bands and any accidental furrows or scratches which may have been left on the specimen through imperfect polishing.

Incidentally, Fig. 16 illustrates the fact that oblique light picks out the boundaries of the crystalline grains, showing that these boundaries are marked by inclined surfaces connecting grains whose faces are at different levels. This is observed also in the etched surface of the metal before straining. The boundaries, which appear dark under vertical light, are bright on one

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side of each crystalline grain when the light falls with grazing incidence from one side; but the sloping surfaces which mark the boundaries between the grains have not the sharply-defined inclination of the slip-surfaces. The lines due to slip-bands on one or more grains will shine out brightly when the light has a particular angle of incidence, and will vanish when the incidence is slightly changed. The boundaries are generally not so bright, but remain visible under a considerable range of incidence.

Figs. 17, 18, and 19 present a striking example of the kaleidoscopic effect under oblique light just referred to. Here the metal is lead, the surface has been obtained by allowing molten lead to solidify on a smooth glass plate, and the metal has been strained by bending. The figures show the appearance under vertical light and also under two different incidences of oblique light. Fig. 20 shows another example of strained lead under vertical light.

In their preliminary notice* of some of the present results (communicated to the Royal Society on March 16, 1899) the authors have applied the name "slip-bands" to the lines developed on metallic surfaces by plastic strain, and in what follows they will be referred to under that name.

So far as the writers' observations go it appears that slip-bands occur in all metals as soon as plastic deformation takes place. They have been observed in specimens of platinum, gold, silver, copper, lead, zinc, tin, cadmium, bismuth, aluminium, iron, and nickel, as well as in steel, brass, bronze, and other alloys. In the case of iron it was proved definitely that under tensile stress the bands appear as soon as the yield-point is passed. For this experiment a flat was polished on the side of a test-piece, which was strained in a 50-ton testing-machine, and the surface was kept under observation, during the application of the stress, by means of a microscope hung from the bar itself.

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* "Experiments in Micro-metallurgy: Effects of Strain," *Proc. Roy. Soc.*, Vol. LXV, page 85. Since writing that paper the authors' attention has been directed to the following remark by Mr. Stead: — "It would appear that there is a tendency for iron to etch out into thin plates, and when such etched specimens are distorted or pulled out for these etched plates to slide over one another." ("On Brittleness Produced in Soft Steel by Annealing," *Journal of the Iron and Steel Institute*, 1898.) It will, however, be shown presently that the development of slip-bands is independent of any previous etching.

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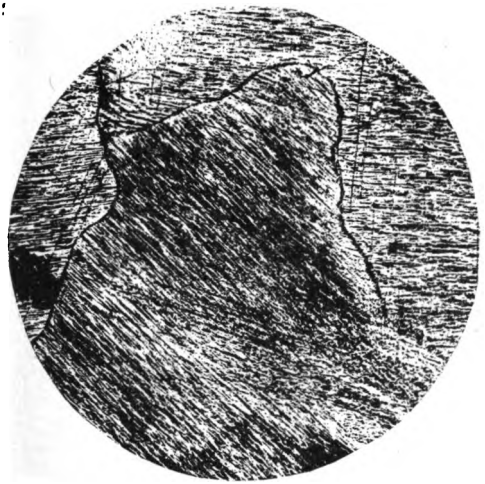

Fig. 17. Glass-cast Lead strained by bending. Magnified 100 diameters. Vertical light.

Fig. 18. Same field as in Fig. 17, seen under oblique light.

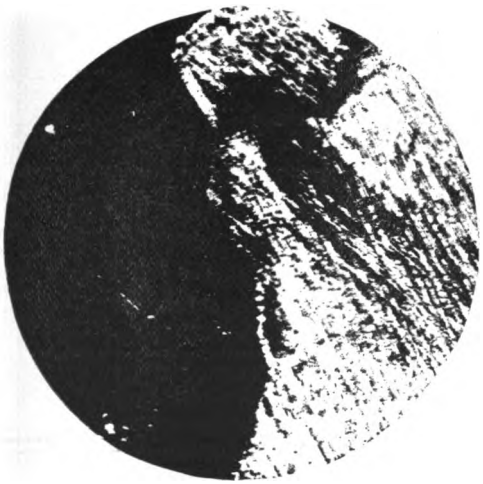

Fig. 19. Same field as in Figs. 17 and 18 after the stage had been turned through about 15°. Oblique light.

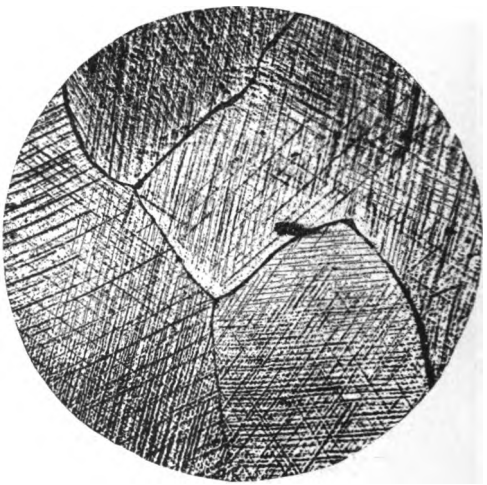

Fig. 20. Slip-bands in Lead. Magnified 100 diameters. Vertical light.

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Slip-bands are developed by all kinds of strain involving permanent deformation of the piece. A microscopic inspection of the surface after straining does not enable us to detect whether the deformation has been caused by tension, compression, bending, or torsion, but the appearance depends very much on the amount of strain that has taken place. The more severely the specimen has been strained the greater is the number of slip-bands developed. After slight straining there is generally only one system of bands to be seen on each crystalline grain, but as many as four such systems intersecting one another may come into view after severe straining. Fig. 20 is a photograph of strained lead, showing four systems of slip-bands.

The general appearance of the slip-bands is different in different metals. In lead they are particularly straight, even under extreme magnification, as is illustrated in Fig. 21. In silver there is a tendency to more wavy outlines, as appears in Fig. 22, but in gold the lines are as straight as those in lead. In iron the lines tend, as a rule, to be rather wavy, and to fork and branch. Examples of slips in this metal have already been given in Figs. 11, 13, and 15; others have been given in the preliminary notice referred to above.*

In gold, lead, and other metals showing straight slip-bands it is easy to distinguish well-marked steps where intersecting systems cross. Fig. 21 is a characteristic example.

In several specimens of lead, prepared by casting against glass, a peculiarity was noticed which forms an apparent exception to the statement made above that slip-bands which shine out under oblique light at one particular incidence do not reappear when the incidence is changed (i.e. the specimen rotated) by 180°. The specimens in question were examined under oblique light with an objective of 16 mm. focus, giving a magnification of 100 diameters. On rotating the stage carrying the specimen the slip-bands on one crystalline grain were found to shine out strongly in two positions, very nearly 180° apart. Under this low magnification it seemed that identical bands were visible in both positions; but on applying a power of 2000 diameters with a combination of "vertical" light and an oblique beam of grazing incidence from an arc lamp it was seen that there were really two systems of parallel slip-bands on the same crystalline grain, and

* *Roy. Soc. Proc.*, Vol. LXV, plates 1–5.

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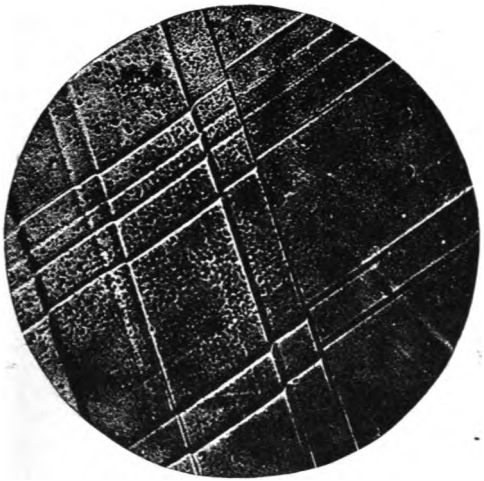

Fig. 21. Slip-bands in Lead.
Magnified 1000 diameters.
Vertical light.

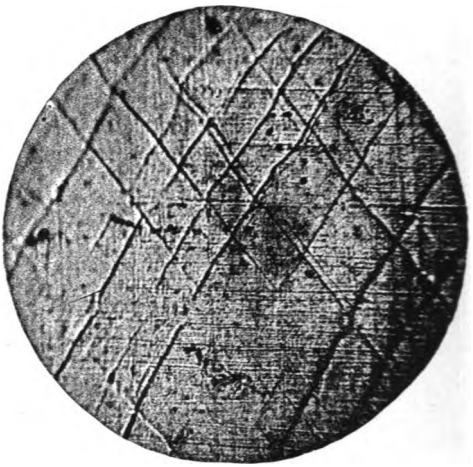

FIG. 22. Slip-bands in Silver.
Magnified 750 diameters.
Vertical light.

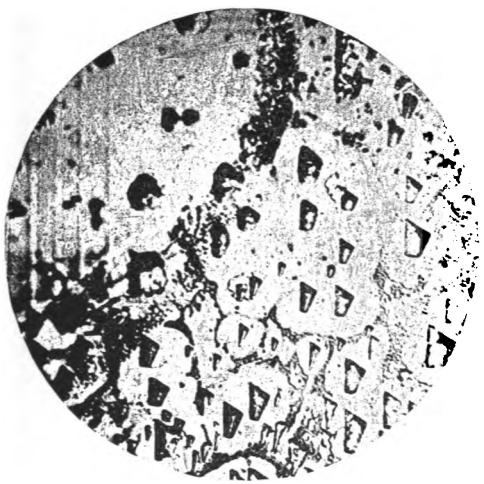

Fig. 23. Geometrical air-pits in glass-cast Cadmium.
Magnified 1000 diameters. Vertical light.

Fig. 24. Etched pits and slip-bands in Iron.
Magnified 1000 diameters.
Vertical light.

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that only one of them was picked out by the oblique light. In the other system of slip-bands the slope of the cleavage surfaces exposed by the slips was inclined away from the source of light, and consequently remained dark. The explanation, then, is that in this grain the crystalline elements are oriented in such a way that the intersection of two sets of cleavage or gliding planes is parallel to the surface, and consequently their traces on the surface are parallel to one another. This seems to be an accidental occurrence, possibly favored by the condition under which the specimen crystallized, namely in contact with cold glass. In many other instances a more or less close approximation to such parallelism has been observed, resulting in systems of slip-bands intersecting at a very small angle; in that case the two positions where slip-bands appear under oblique light are nearly, but not exactly, 180° apart. Seen under vertical light, with low magnification, such intersecting systems produce the appearance which is exemplified in the central grain in Fig. 17.

The relation of slip-bands to the geometrical air-pits in cadmium is illustrated by the photograph, Fig. 23. In this example the metal was cast against glass in such a way as to produce air-pits, and was then strained sufficiently to develop slip-bands. It appears that the slip-bands are always parallel to one side of the geometrical pits — the two phenomena thus confirming the views which have been advanced above as to the crystalline structure of the metal. Fig. 24 shows the relation of slip-bands to geometrical etched pits in iron. It will be observed that the slips are not generally parallel to a side of the etched figures, but in specimens that have been more severely strained than those here illustrated, one set of slip-bands in each grain is generally found to be parallel to one side of the etched pits, while the other systems intersect these sides diagonally. The observations point to the conclusions that in iron slipping occurs most readily along the octahedral planes, although slips parallel to the sides of the cubical crystals are also found.

The development of slip-bands in strained metal throws what appears to us to be a new light on the character of plastic strain. Plasticity is due to the occurrence of these slips. When metals are strained beyond their limit of elasticity the deformation occurs through sliding over one another of the elementary portions of which each crystalline grain is built up.

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The sliding which gives rise to slip-bands is of finite amount, and occurs at a limited number of places. "Flow" or plastic strain in metals is not a homogeneous shear such as occurs in the flow of a viscous fluid, but is the result of a limited number of separate slips.

The conception that metals adapt themselves to the new shapes imposed upon them when they undergo plastic deformation by means of slips along cleavage or gliding planes within each crystalline grain, leads naturally to the supposition that the crystalline elements themselves undergo no deformation in this process. The portions of the metal between one surface of slip and the next may remain undeformed, except elastically, under all stresses. The effect of a stress sufficient to produce plastic strain is analogous to that of a tractive force overcoming the static friction between two surfaces.

If we assume that plastic strain takes place solely by these slips, it follows that the ultimate crystalline elements should always remain parallel to themselves. The orientation of the elements would remain uniform throughout the mass of one grain, however much the outline of that grain were distorted by slips occurring within it. In other words, the crystalline structure of a metal should survive even the severest strain.

This conclusion is borne out by the fact that in metals which have been much strained we find evidence of crystalline structure similar to that which is found in unstrained metal.

Taking this evidence in the same order as before, we would refer first to the appearance which is presented in severely strained metal, by a polished and etched section when seen under oblique light. We have examined a bar of fine Swedish iron (kindly supplied by Messrs. Edgar Allen and Co.), which had been for the purpose of these experiments rolled in the cold state from a diameter of three-quarters of an inch to a diameter of half an inch, and had not been subsequently heated. A longitudinal section of this cold-rolled bar showed a great lengthening of the crystalline grains in the direction of rolling, and even in the transverse section it was obvious that the grains had been much distorted, though there was no direction of predominant length. Under oblique light both sections (longitudinal and transverse) exhibit the effect described above for unstrained metal — the grains are distinguished from one another by differences of

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brightness, which vary when the incidence of the light is altered, and this brightness is uniform over the entire surface of each grain. As in the case of unstrained metal, we regard this as evidence of the uniform orientation of the crystalline elements throughout each grain. Fig. 25 is a photograph under oblique light of a specimen of this bar cut transversely, and polished and etched. It illustrates the uniform brightness of each grain, due to uniform orientation. Similar characteristics have been observed in many other specimens of severely strained metal.

Another line of evidence in proof of the persistence of crystalline structure after severe straining is afforded when a polished specimen of, say, cold-rolled iron is subjected to a slight further strain. The slip-bands appear as they would have done had the specimen never been strained before. The general features are much the same as in annealed metal, but they show on the whole a greater tendency towards sudden steps and branches. This difference in the character of the slips may be connected with the well-known fact that such strained material is considerably harder, in the sense of having a higher yield-point and less capability of plastic deformation than it shows in the virgin or annealed state, and also with our own observation that the slip-bands are much more straight and regular in very plastic metals, such as lead, gold, and copper, than in harder metals like iron and nickel. The mere fact that finite slips occur at intervals throughout the grain implies that it is easier for sliding to take place over certain surfaces than it is over others. The surface on which uniform slip occurs is not necessarily plane; it may be the trace of a straight line moving parallel to itself; a line which is in the direction of slip and always lies parallel to one of the cleavage planes. For convenience in argument we may for the moment assume that the surfaces of easiest sliding are determined by some accident, such as the presence there of minute layers of some impurity, such as occluded gas. If, as the straightness of the slip-bands seems to indicate, these surfaces are true planes in plastic metals like lead or gold (so far as each individual grain is concerned), sliding might take place in two directions on each of these planes. But when sliding has once taken place, the intersecting layers of impurity would no longer be distributed over planes, and further sliding on transverse planes would necessitate the starting of fresh slips in surfaces that had no special tendency

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Fig. 25. Polished and etched sections of cold-rolled Swedish Iron.
Magnified 45 diameters. Oblique light.

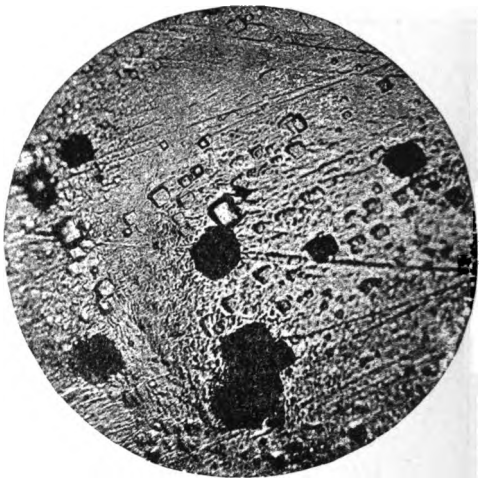

Fig. 26. Distorted grains of a transverse section of cold-rolled Swedish Iron, also showing etched pits.
Magnified 200 diameters. Vertical light.

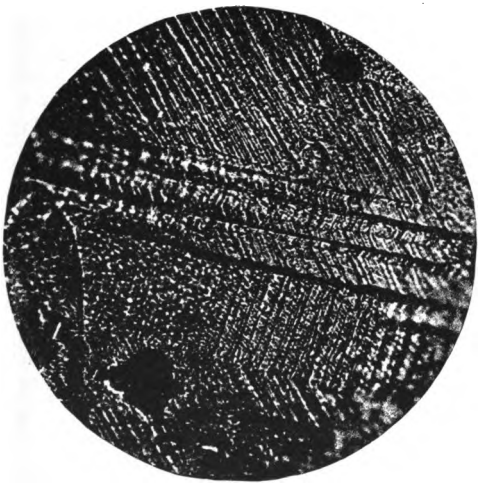

Fig. 27.

Fig. 28.

Slip-bands in twin crystals of Copper.
Magnified 1000 diameters. Vertical light.

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to facilitate sliding. This suggests how such a metal may be hardened by previous straining; and how, also, the slip-bands formed on re-straining a piece of metal hardened in this way would be less straight and more liable to sudden steps and branches than those that are found in the virgin material; the slips would, as far as possible, follow the old surfaces of easy sliding, but these would now be stepped instead of plane as before.

A striking proof of the persistence of crystalline structure in metals which have been submitted to severe distortion is found in the existence of geometrical etched pits. These are readily developed in sections cut from cold-rolled iron, and they differ in no way from the etched pits developed in the virgin material; like these, they appear as similar and similarly oriented geometrical figures over the face of each grain Fig. 26 is a photograph under 800 diameters of a portion of grain of cold-rolled Swedish iron referred to above, with an outline so unlike those found in unstrained metal that its form is evidently due to violent distortion in the process of rolling. The face of this grain is covered with minute etched pits, and an examination of these under high powers shows that they have preserved their similarity of shape and orientation in spite of the violent distortion which the grain, as a whole, has undergone.

From the various lines of evidence here indicated we conclude that the characteristic crystalline structure of metals is not destroyed by strain, no matter how severe, and that plastic deformation occurs by means of slips along the cleavage or gliding planes of the crystalline grains, the crystalline elements which build up each grain remaining unaltered both as to shape and orientation. This statement, however, is subject to the following qualification. We have found in certain metals, notably copper, gold, silver, lead, cadmium, tin, zinc, and nickel, that twin crystals are liable to be developed by straining. Hence in such cases it is not exact to say that straining produces no change in the orientation of the crystalline elements, for twinning implies a rotation of one group of elements with respect to the rest through a definite angle. The twinning which we have found in many strained metals corresponds to the twinning observed in calcite by Baumhauer and Reusch, and subsequently produced in isolated crystals of antimony and bismuth by Mugge.*

* See P. Groth's *Physikalische Krystallographie.* Voss, Leipzig.

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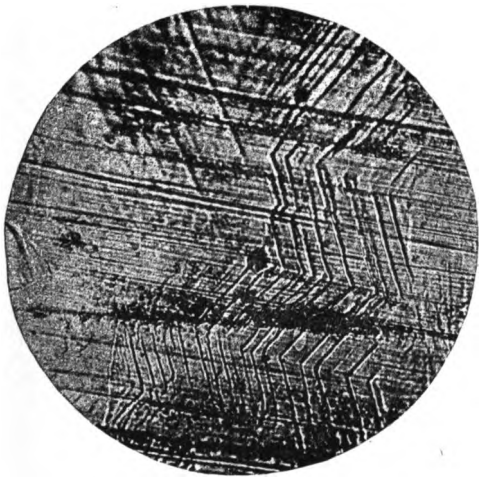

Fig. 29. Slip-bands in twin crystals of Copper.
Magnified 1000 diameters. Vertical light.

Fig. 30. Slip-bands in twin crystals of Gold.
Magnified 200 diameters.
Vertical light.

Fig. 31. Slip-bands in twin crystals of Gold.
Magnified 45 diameters.
Oblique light.

Fig. 32. Slip-bands in twin crystals of cold-hammered Copper.
Magnified 1000 diameters. Vertical light.

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Baumhauer found that by forcing a knife blade into a crystal of calcite at the proper angle a portion of the crystal could be made to swing over into the twinned position. This implies a corresponding change of orientation in the crystalline elements. The experiment may be said to afford an example of plasticity in a crystal, but it is not entirely analogous to the plasticity by pure sliding which is found in iron and in most other metallic crystals. In the process of twinning by strain there is both slip and rotation of the elements.

The existence of twin crystals in certain metals became apparent when systems of slip-bands were found like those shown in Figs. 27, 28 and 29. These are photographs from specimens of copper. They show that certain of the crystalline grains are crossed by twin lamellæ, the twin planes being defined by a sudden change in the direction of the slip-bands. Where several such twin lamellæ occur in one crystalline grain, we have a periodic structure with alternate systems of parallel slip-bands. The change of orientation in passing from one lamella to another is constant. It is clear that in these examples we have true cases of twin crystallisation. An example of twinning in gold, as seen under vertical light, is given in Fig. 30, and Fig. 31 is a photograph of twins in gold, seen under oblique light.

The question arose whether these twin crystals were a feature in the primitive crystallisation of the metal, or whether they were subsequently produced as a consequence of strain. They were first seen in wrought copper (namely in a piece of rolled plate), which had been raised to a bright red heat before polishing. We next examined specimens of copper, gold, silver and lead, each in the cast state, and in none of these found any appearance of twinning. For the purposes of this examination only a slight strain was applied. The same pieces were then wrought, that is to say they were severely strained (namely, by cold hammering) and they were again examined both before and after annealing at a red heat. The result showed that the violent strain produced by working the metal had developed twins in specimens where none could be seen before, and that the twins were still found in the wrought specimens after annealing. Fig. 32 is a photograph of a "twin" in cold-hammered copper (not heated after hammering); incidentally it illustrates the persistence of crystalline structure after violent deformation. Still more striking in this respect

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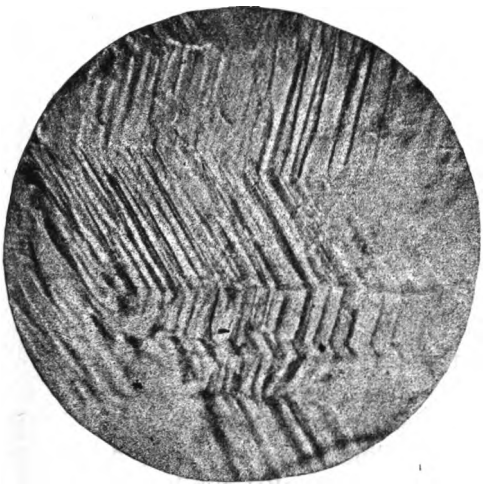

Fig. 33. Slip-bands in twin crystals of Sheet Lead.
Magnified 1000 diameters. Vertical light.

Fig. 34. Slip-bands and twinning in Nickel.
Magnified 1000 diameters.
Vertical light.

Fig. 35. Elongated crystalline grains in Cadmium cast on a sloping surface of glass.
Magnified 100 diameters. Vertical light.

Fig. 36. Glass-cast surface of Cadmium, showing double system of boundaries.
Magnified 100 diameters. Vertical light.

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is the appearance shown in Fig. 33. The specimen in this instance was an ordinary piece of plumber's sheet lead; the surface was scraped bright with a knife, and was then squeezed against a piece of plate-glass in a vice, thus producing a beautiful surface. The specimen was then very slightly bent in the fingers to develop slip-bands, and on examination under a high power it showed the appearance reproduced in the photograph.

Twinning as revealed by slip-bands, has been observed in nickel; in this case the specimen was a virgin casting, but the twin (shown in the photograph, Fig. 34) was observed only after very severe straining, and was, in all probability, produced by that straining.

In zinc, tin, and cadmium twins either occur very freely in the cast metal, or else are very readily produced by the slight strain which is applied to develop slip-bands. Surfaces of these metals, produced by casting against glass, show twin bands even under fairly low powers when the specimen is slightly bent; the twin bands then appear as shaded bands running across the crystalline grains. Very frequently the twin bands run on continuously across two or more grains, with more or less change in direction when they cross a boundary.

A specimen of cadmium showing this feature is photographed in Fig. 35. This particular specimen presents another peculiarity; it was prepared by casting against glass, and in this instance the glass surface was intentionally given a considerable slope, with the result that the metal solidified in a long strip while it was running down the glass. On examining the under face of this strip two modes of crystallisation were observed. Part of the surface there showed a very small structure with no direction of greatest length. On another part there were large grains very considerably longer in the direction of the length of the strip than in a transverse direction, passing across from one elongated grain to the next with only a slight change in direction. The photograph, Fig. 35, is taken from an area showing these long grains. When the piece was strained by bending, twin lamellæ appeared in a more or less transverse direction. The twin band in one grain is associated with a twin band in a neighboring grain, the bands being continuous except for a change of direction as they pass from grain to grain.

We have observed twinning in gold, silver, copper, lead,

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nickel, zinc, tin, and cadmium. It does not appear to occur in iron.

The facility with which most metals undergo twinning as a consequence of strain shows that there are in general two modes by which plastic yielding takes place in an aggregate of crystals. One is by simple slips, where the movements of the crystalline elements are purely translatory, and their orientation is preserved unchanged. The other is by twinning, when rotation occurs through an angle which is the same for each molecule in the twinned group. Both modes are often found in a single specimen of metal, and even in a single crystalline grain. Thus, in gold or copper, it is very usual to find, on examining a strained specimen that one portion of a grain is covered with simple slip lines, while another portion of the same grain shows one or more lamellæ which are twinned with respect to the rest of the grain.

On surfaces prepared by casting against glass, particularly with cadmium, but also with zinc and tin, a curious feature often occurs which is closely associated with the facility these metals show in developing twins. The appearance in question is that of an apparent duplication of the inter-granular boundaries, as seen in the cadmium casting, Fig. 36. The second system of boundaries consists, like the first, in polygonal markings, and has such an obvious relation to the first that it almost appears as though the upper layer of crystalline grains were transparent, and that we were seeing their lower edges. To decide as to which of the markings on the surface constituted the true surface boundaries, two methods were available: etching with an acid and slightly straining the specimen so as to develop the slip-bands. The latter method is much the more instructive, and its results are confirmed by the etching process. When a specimen having this characteristic was strained, it was seen at once that some of the apparent boundaries were consistently ignored by the slip-bands, the others being the real junction lines of the grains. But the true nature of the pseudo-boundaries comes out on examining them under a high power. Although under low powers there is no obvious difference in definition between the genuine and pseudo-boundaries, under greater magnifications it becomes impossible to focus the pseudo-boundaries at all — they are seen to be more or less ill-defined slopes or changes of level, whereas the

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real boundaries are sharply defined. In general the real boundaries show some accumulation of gas bubbles along them, and they are never crossed by slip-bands. The pseudo-boundaries are found to consist in small variations of level in the surfaces of the grains in which they occur. Fig. 37 is a high-power photograph of a set of real and pseudo-boundaries showing slip-bands.

It will be noticed that on the two sides of a real boundary the slip-bands are independent of one another, whereas the slip-bands cross a pseudo-boundary with only a slight change of inclination, which is to be ascribed to the fact that the surface under examination is not a true plane. There is a slight slope on each side of the pseudo-boundary, and the lines are consequently more or less inclined to one another. Again, as we have noticed in other examples the slope is not as a rule constant and hence the lines are slightly curved.

An explanation of this appearance of pseudo-boundaries is, we think, to be found in the strains set up by contraction on cooling. If we suppose the outer layer of crystals to cool more rapidly than the inner ones, the resulting contraction will drive the projecting edges of the lower layer into the outside grains and thus cause slight local deformation, which will project itself on the surface, probably by means of twin bands running through the grains and appearing on the surface. The effect resembles that of a Japanese "magic" mirror, in which slight inequalities of the surface, corresponding to a pattern behind, cause light reflected from the mirror to produce an image in which a ghost of the pattern may be traced.

The foregoing conclusions refer to experiments on pure or nearly pure metals. We have also examined the effects of strain on various alloys. The micro-structure of alloys has received attention at the hands of most of the workers already named, especially Behrens, Charpy, Guillemin, Osmond, Roberts-Austen, and Stead. Our observations have been directed towards supplementing theirs, in respect particularly of the effects of strain.

The experiments on iron were extended to certain steels. In very mild steel slip-bands can be readily observed in what are generally called the "ferrite" areas, which remain white after light etching. This is shown in Fig. 38. The first effect of strain is to develop the inter-granular junctions in these white

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Fig. 37. Real and pseudo-boundaries as indicated by slip-bands. Magnified 1000 diameters. Vertical light.

Fig. 38. Strained Mild Steel, showing pearlyte patches and slips in ferrite areas. Magnified 1000 diameters. Vertical light.

Fig. 39. Glass-cast and etched surface of Lead Tin, eutectic. Magnified 750 diameters. Vertical light.

Fig. 40. Glass-cast and strained (but unetched) Lead Tin, eutectic. Magnified 750 diameters. Vertical light.

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areas, then more severe straining makes the slip-bands appear. In steels containing larger proportions of carbon, the scale of the granular structure of the "ferrite" diminishes and the slip-bands become correspondingly minute, requiring the highest powers of the microscope for their observation. We have not been able to observe anything of the nature of slip-bands in the dark or "pearlyte" areas of steel, but the correspondence which has been recognized by Osmond to exist between the structure of "pearlyte" and that of typical eutectic alloys, taken with facts to be described below, points to the possibility that "pearlyte" may also yield plastically by slipping.

Slip-bands have also been observed in various specimens of brass and bronze.

The behavior of eutectic alloys under plastic strain is of special interest, because these bodies apparently differ so widely in structure from pure metals. Our observations have been made on the eutectics of lead-tin, copper-silver, and lead-bismuth. The micro-structure of such eutectics has been described by Osmond.

Fig. 39 is from a specimen of lead-tin eutectic kindly prepared for us by Messrs. Heycock and Neville; the surface was obtained by casting against glass, and was lightly etched with a 1 per cent solution of nitric acid. Figs. 40 and 41 illustrate the most obvious effect of strain on such structures; the surfaces have not been etched, the differentiation of the two constituents by differences of level being here entirely due to strain. It will be observed that the scale of this structure is similar to that of the slip-bands seen in pure metals, and examination of strained specimens shows that plastic yielding is associated with slips occurring between layers of the two constituents. A close examination of strained specimens has enabled us to detect slip-bands in the light-colored constituent. By adopting the device of slow cooling, which has led to such excellent results in the hands of Messrs. Heycock and Neville, we have succeeded in producing specimens of eutectics in which the characteristic structure is developed upon a much larger scale. Fig. 42 exemplifies this in the eutectic of bismuth and lead, and shows slips which occur in the white constituent as a consequence of straining. This photograph illustrates a feature very characteristic of eutectic alloys; a parallel system of slip-bands extends over many patches of the white constituent, thus pointing to the fact that the

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Fig. 41. Glass-cast and strained (but unetched) Lead Tin, eutectic.
Magnified 750 diameters. Vertical light.

Fig. 42. Slowly cooled Lead-bismuth, eutectic, etched and strained, showing slip-bands.
Magnified 1000 diameters. Vertical light.

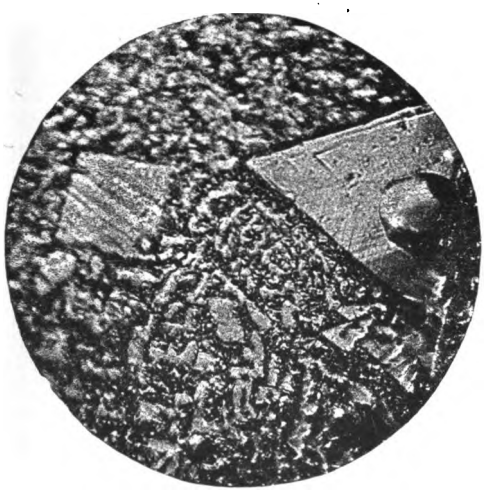

Fig. 43.

Fig. 44.

Slip-bands in Crystallites found in Lead-bismuth Alloy.
Magnified 1000 diameters. Vertical light.

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crystalline elements are similarly oriented throughout considerable areas of at least one of the two constituents of the alloy. This suggests that the alloy as a whole has comparatively coarse granular structure, and the same conclusion is borne out by observing the general character of a surface under lower magnifications (such as 100 diameters), when its structure is revealed either by straining or etching. The surface is then seen to be divided into rather large more or less polygonal areas, each covered with a system of ribs radiating from one point, giving an appearance which resembles roughly the ribs of an umbrella. Fig. 40 incidentally shows the boundary of two such areas.

In the course of experiments on the lead-bismuth eutectic, specimens were obtained showing comparatively large isolated crystallites. When the piece was strained these crystallites were found to exhibit slip-bands. Examples are given in Figs. 43 and 44. These are interesting as showing the development of slip-bands in bodies which are evidently fully developed crystals, even as to external form.

A study of the micro-structure of alloys suggests a possible explanation of the peculiarities they present in regard to variation of electrical conductivity with temperature. The two constituents may behave individually as pure metals in this respect, but if their coefficients of expansion are different the closeness of the joints between them will depend on the temperature. Thus, if the more expansible metal exists as plates or separate pieces of any form within the other, the effect of heating will be to make the joints between the two conduct more readily, with the result of reducing the increase of resistance to which heating would otherwise give rise, and in extreme cases with the effect even of producing a negative temperature coefficient.

Reviewing the general results of the experiments, we consider that they establish the view that the structure of metals in general is crystalline, and remains crystalline when the form of the metal is altered by strain, plastic yielding being due to slips on cleavage or gliding planes within each individual crystalline grain, and partly (in some metals) to the production of twin crystals. In a pure metal, when straining is carried far enough to produce fracture, the crystalline grains suffer cleavage, and the cleavage surfaces thus developed give to the fracture its characteristically crystalline appearance. In impure metals fracture

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may occur through the parting of grains from one another at their boundaries. In both cases, however, the plastic yielding which precedes fracture takes place by slips in the manner we have described.

In conclusion we should like to express our indebtedness to Sir W. Roberts-Austen, Mr. T. Andrews, and Professor Arnold, for giving us at the outset of our work the benefit of their large experience in preparing specimens of metals for microscopic examination. Messrs. Heycock and Neville and Mr. A. Hutchinson have assisted us materially by various suggestions, and by supplying specimens for examination. We have also to thank Mr. Andrews, Mr. Stead, Mr. Hadfield, Professor Hicks, and Messrs. Edgar Allen & Co. for special specimens of iron.

The work described in this paper was carried out in the Engineering Laboratory at Cambridge.

THE EFFECT OF HEAT-TREATMENT UPON THE PHYSICAL PROPERTIES AND THE MICROSTRUCTURE OF MEDIUM-CARBON STEEL.*

By ROBERT GORHAM MORSE.

I. Introductory.

THIS paper presents the results of an investigation made in the metallurgical laboratory of Columbia University, New York City.

The object of the investigation was to determine how the tensile strength, the elastic limit, and the size of the grain, of a pure medium-carbon steel are affected by heating to successive temperatures between 500° and 1300° C., with slow cooling therefrom.

Since Dr. Sorby's first investigations into the microstructure of steel, the study of the subject has been continued by Professor Martens, Mr. F. Osmond, Professor Arnold, Professor Howe, Mr. A. Sauveur, Mr. J. E. Stead, and many others. Among

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* Read at the September, 1899, meeting of the American Institute of Mining Engineers.

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many valuable and interesting papers I mention the following as having a special bearing upon this investigation.*

Microstructure of Steel, by Albert Sauveur, *Trans.,* xxii., 546 (1893); *Microscopic Metallography,* by F. Osmond, *Trans.,* xxii., 243 (1893); *The Microstructure of Steel and the Current Theories of Hardening,* by Albert Sauveur, *Trans.,* xxvi., 863 (1896); *The Crystalline Structure of Iron and Steel,* by John Edward Stead, *J. Iron and Steel Inst.,* 1898.

II. Statement of Experiments.

Through the kindness of Mr. Lilienberg, New York City, round rods ¾ of an inch in diameter were obtained, which had been rolled from one ingot. The analysis of the steel was as follows:

C, 0.343; Si, 0.027; Mn, 0.221; P, 0.0266; S, 0.0037 per cent.

Pieces 1 ft. long were heated to the following temperatures:

500°C	680°	800°	1200°*x*
600°*x*	687°	900°*x*	1300°
646°	700°	1000°	
654°	725°	1100°	

Pieces were also heated to the respective temperatures marked *x* and held there for periods of one-half hour, one hour, and three hours. Heatings were made in a gas-furnace, the bars being enclosed in a wrought-iron tube to equalize the temperature, and to prevent oxidation. As soon as the desired temperatures had been reached, the bars were removed from the furnace and imbedded in lime to ensure a slow and uniform rate of cooling. A Le Chatelier thermo-electric pyrometer was used to record the temperatures. The bars were then tested for tensile strength and elastic limit in a Riehlé testing-machine. The latter was determined only by the drop of the beam. The figures are therefore only approximate, as the drop was not always well-marked. After testing, a section was cut off the end of each specimen, polished, etched with nitric acid, and examined under the microscope. Photomicrographs were then taken with perpen-

* A full list of the papers which have appeared on the subject is given in *The Metallographist,* April, 1898, page 168.

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Fig. 1.

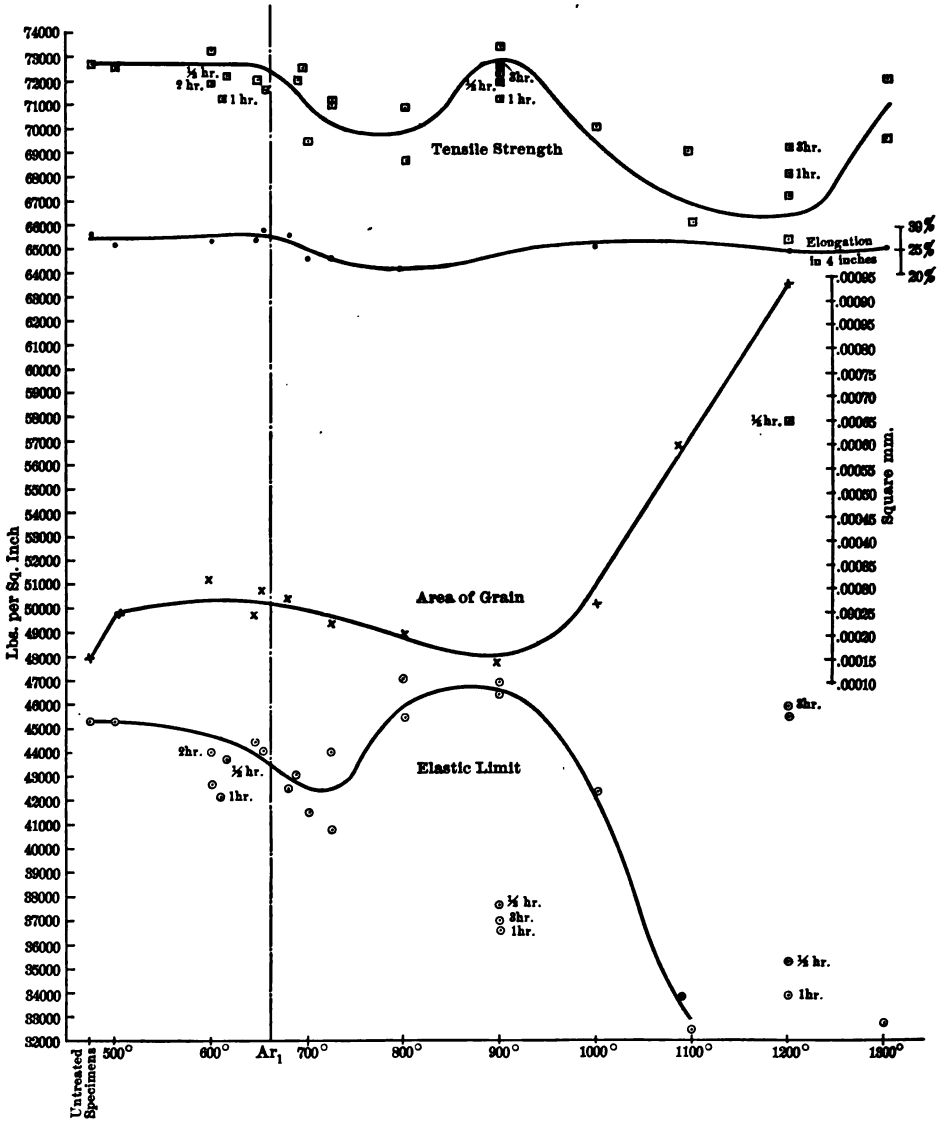

Diagram showing the effects of heat-treatment upon Steel, as described.

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dicular illumination. The ferrite appears light and the pearlyte dark.

The area of the grains of ferrite was next determined in the following manner. The number of grains in a measured area on a photograph was counted, taking the average of as many different areas as possible. Reducing this area to actual size by dividing by the area-magnification, we can determine the average area of a grain of ferrite, if we know what percentage of the measured area is ferrite, by dividing by the number of grains counted. Now the theoretical percentages by weight of ferrite and pearlyte in any carbon-steel can be calculated. Following the method described by Mr. Sauveur in his paper of 1896 (mentioned above), steel of 0.343 per cent carbon is found to contain about 58 per cent ferrite (the percentage of ferrite would be slightly larger in a very pure steel, and slightly smaller in a less pure one).

Assuming the specific gravities* of ferrite and pearlyte to be 7.90 and 7.84 respectively, the percentage of ferrite by area is found to be 58.17 per cent. Therefore:

$$\frac{\text{Measured area} \times 0.5817}{\text{Number of grains counted}} = \text{Average area of grain.}$$

Photographing with a higher magnification would have been interesting in a number of instances. Higher-power objectives were tried, but without success, as the field could not be properly focussed.

The results of our observations are plotted on Fig. 1, in which the figures for tensile strength, elastic limit, elongation, and area of the grain are taken as ordinates, and the temperatures as abscisses.

Fig. 2 exhibits the cooling curves which determine the critical point Ar_1, shown in this case to be about 661° C.

Figs. 3 to 23, inclusive, are reproductions of the microphotographs. With one exception, the engravings are all on the same scale as the photographs. Fig. 16 was in photograph magnified 35 diameters, but was reduced in engraving to 25.

* *Metallurgy of Steel*, H. M. Howe, page 257.

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Tabulated Results, Shown Graphically in Fig. 1.

Maximum Temperature. Degrees C.	Elastic Limit. Pounds per sq. in.	Reduction of Area. Per cent.	Tensile Strength. Pounds per sq. in.	Elongation in 4 in. Per cent.	Area of Grain. Sq. mm.	Magnification of Photo. Diameters.	Fig. No.
500 . . .	45,340	50	72,600	26	0.00024	137	5
600 . . .	42,650	48	73,230	27	0.00031	137	6
600 for ½ hr.	43,670	50	72,210	27	. . .	. .	. .
600 " 1 "	42,130	51	71,360	29	. . .	. .	. .
600 " 2 "	44,020	52	71,970	27	0.00028	137	9
646 . . .	44,430	49	72,060	26	0.00024	137	7, 8
654 . . .	44,030	52	71,610	29	0.00029	137	10
680 . . .	42,440	50	72,060	28	0.00027	137	11
687 . . .	43,000	51	72,590	27	. . .	. .	. .
700 . . .	41,470	53	69,550	23	. . .	. .	. .
725 . . .	40,820	53	71,090	23	0.00023	137	12
800 . . .	44,530	52	68,700	21	0.00020	137	13
900 . . .	46,460	54	73,430	27	0.00015	137	14
900 for ½ hr.	37,700	49	72,070	27	. . .	137	15
900 " 1 "	36,650	44	71,310	24	. . .	137	16
900 " 3 "	36,980	48	72,700	26	. . .	137 35	17 18
1000 . . .	42,300	47	70,920	26	0.00026	137	19
1090 . . .	33,800	44	69,210	26	0.00029	137	20
1200 . . .	45,460	45	67,340	25	0.00093	137	21
1200 for ½ hr.	35,380	42	56,890	23	. . .	137	22
1200 " 1 "	33,810	47	69,280	25	. . .	137	23
1200 " 3 "	46,010	46	68,230	23	. . .	137	24
1300 . . .	32,710	44	68,720	26	. . .	137 137 35 10	25 26 27 28

Untreated Specimens.

. . . . .	46,680	47	72,730	23	0.00015	137	3, 4
. . . . .	44,250	52	73,090	28	. . .	. .	. .
. . . . .	44,610	52	72,420	24	. . .	. .	. .
. . . . .	45,720	50	72,480	26	. . .	. .	. .

III. Effect of Heat-Treatment on the Microstructure.

1. *Slow Cooling Immediately from T max.* — An examination of the photographs will show that both the size and the shape of the grain change under certain conditions. The specimens shown in Figs. 3 to 12, inclusive, have all been cooled slowly from a temperature not exceeding 900° C. The grains of ferrite

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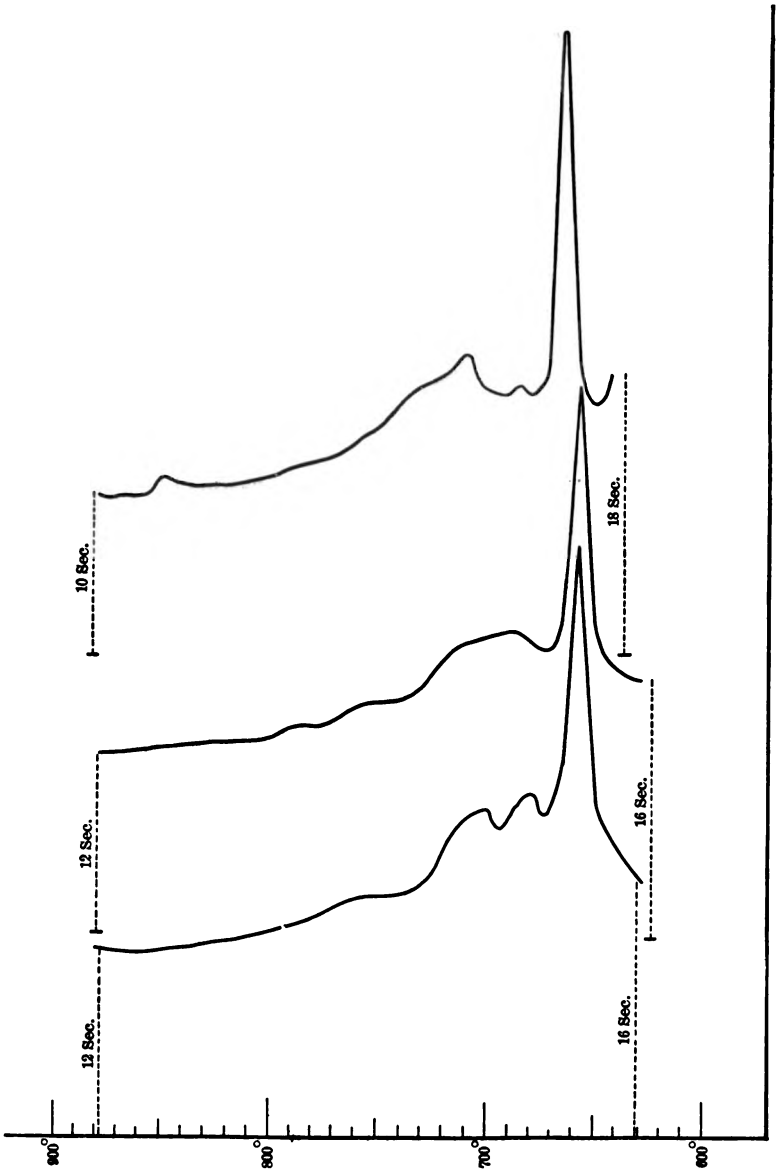

Fig. 2. Cooling curves to determine the critical point, Ar_1.

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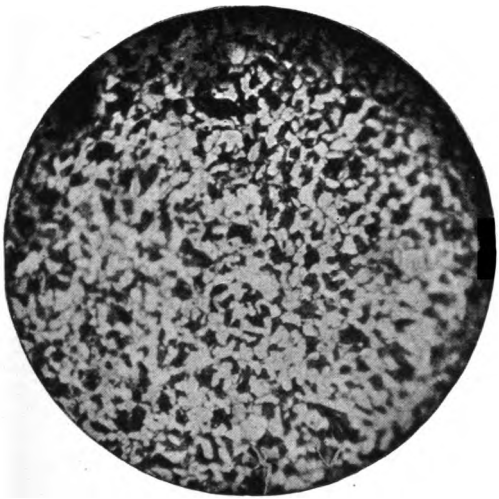

Fig. 3. Section of rod, before special heat-treatment. 137 diameters.

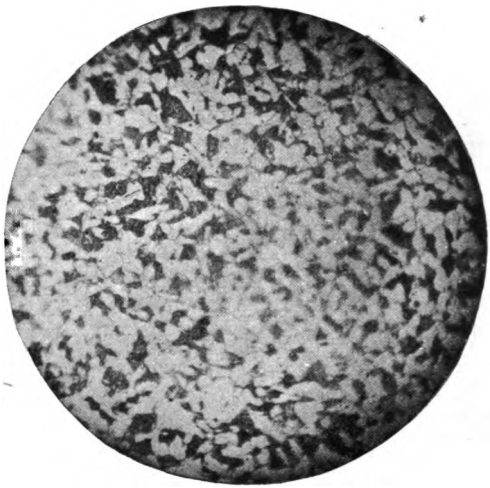

Fig. 4. Section of rod heated to 500° C., and immediately allowed to cool. 137 diameters.

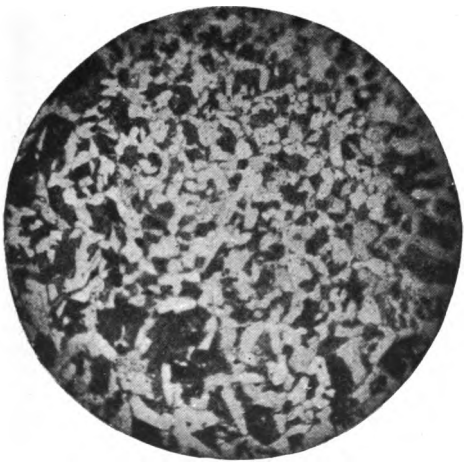

Fig. 5. Section of rod heated to 600° C., and immediately allowed to cool. 137 diameters.

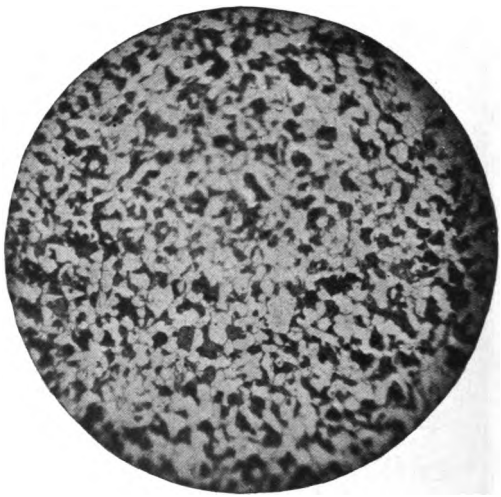

Fig. 6. Section of rod heated to 646° C., and immediately allowed to cool. 137 diameters.

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in all these have the same general appearance; though of irregular shape, they are more or less equiaxed. The pearlyte is in the form described by Osmond and Werth as simple cellular. In Fig. 4 (heated to 500°) the grain is considerably larger than in the untreated specimen (Fig. 3). This is probably due to the fact that the rolling which the bar underwent in process of manufacture squeezed out the grains longitudinally, making them smaller in transverse section. (This would seem to indicate that the finishing temperature of the rolling was very low, at least below 500°; otherwise the grain would have had a chance to draw itself into a rounder shape, as appears in Fig. 5. It would be interesting in this connection to polish and examine a section cut longitudinally from the untreated bar.) The area of the grain seems to be fairly uniform until the critical point 661° is reached. Here it begins to grow steadily smaller, reaching a minimum at 900° (Fig. 12). From this point on the appearance of the grain begins to change. Passing to 1300° (Fig. 23), we find that the form has altogether changed. For example, compare Fig. 8 with Fig. 23. In the former, the ferrite appears in distinct grains of irregular, but more or less rounded, shape, imbedded in a mass of pearlyte, giving rise to the term "simple cellular." In Fig. 23, on the other hand, a more complex condition seems to exist. The ferrite grains have grouped themselves, some into bands and some into thin lamellæ or scales, so that it is quite impossible to count them. The photographs of the specimens heated to 1000°, 1100° and 1200° (Figs. 17, 18, and 19) show the gradual growth of the ferrite grain, and the transition of the ferrite from the simple cellular to the lamellar form. The change in the ferrite grain is gradual from 900° to 1200°; but between 1200° and 1300° it seems to have lost completely its structure in separate grains, and formed itself into continuous bands or ramifications striking into, or sometimes through, the fairly well-defined grains of pearlyte. Our curve for the size of the ferrite grain must, therefore, necessarily stop at 1200°.

2. *Holding at T max. for Periods of Time.* — Figs. 12 to 15, inclusive, show what a remarkable change has taken place in the form of the grain upon exposure to the same temperature for periods of time. At 1900°, in half an hour (Fig. 13) the granular form of the ferrite has almost disappeared, though the

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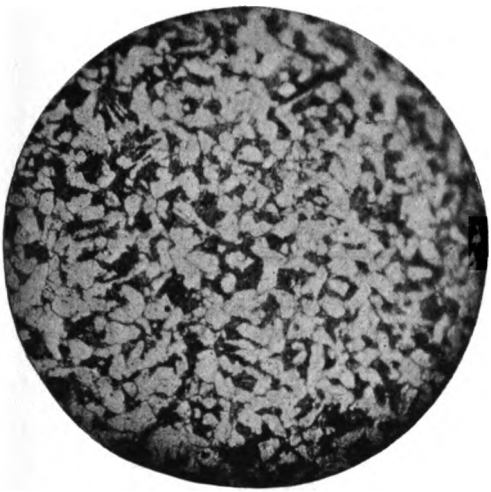

Fig. 7. Section of rod heated to 600° C., and held at that temperature for two hours before cooling. 137 diameters.

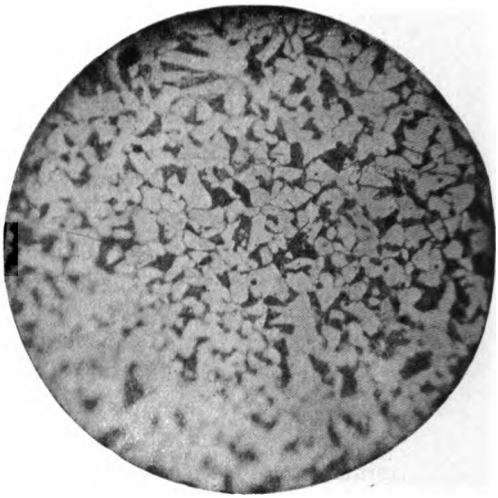

Fig. 8. Section of rod heated to 654° C., and immediately allowed to cool. 137 diameters.

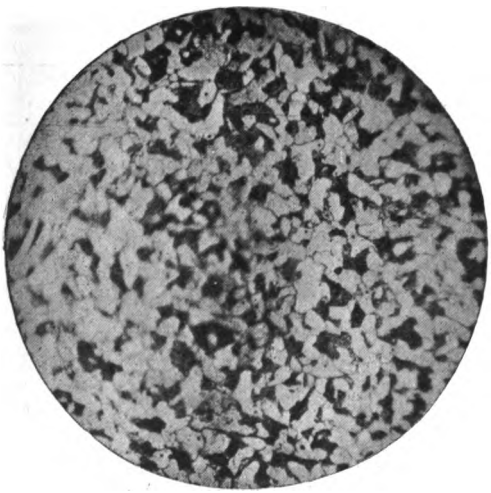

Fig. 9. Section of rod heated to 680° C., and immediately allowed to cool. 137 diameters.

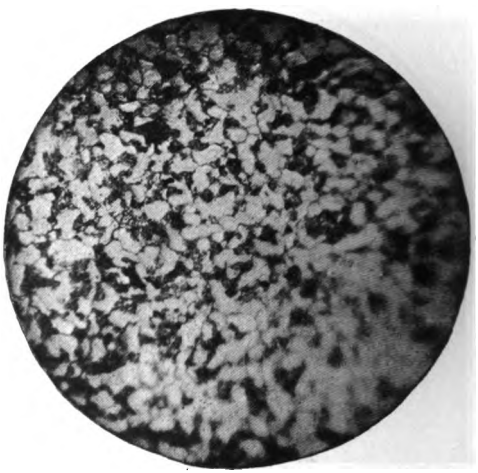

Fig. 10. Section of rod heated to 725° C., and immediately allowed to cool. 137 diameters.

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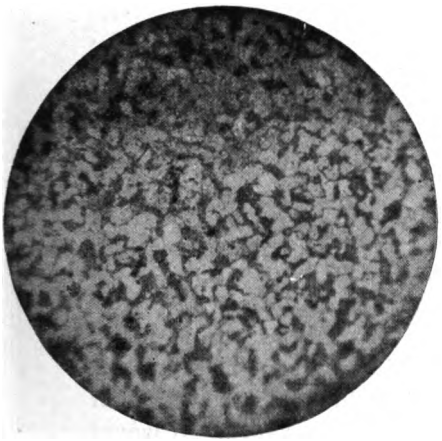

Fig. 11. Section of rod heated to 800° C., and immediately allowed to cool. 137 diameters.

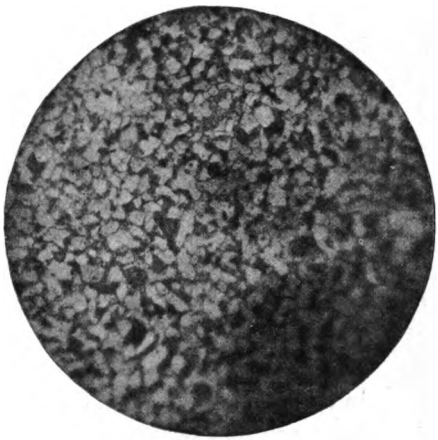

Fig. 12. Section of rod heated to 900° C., and immediately allowed to cool. 137 diameters.

Fig. 13. Section of rod heated to 900° C., and held at that temperature for half an hour before cooling. 137 diameters.

Fig. 14. Section of rod heated to 900° C., and held at that temperature for one hour before cooling. 137 diameters.

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pearlyte (at least so far as this magnification shows) has not begun to form regular grains. In an hour (Fig. 14) the ferrite has become much banded or ramified, and the lamellæ of the pearlyte have grown to a plainly visible size. At the end of three hours (Fig. 15) the ferrite has entirely lost its granular form, and the pearlyte has taken the shape of more or less regular grains surrounded by bands and pierced by striations of ferrite.

The same features are shown still more strikingly in Figs. 19 to 22, inclusive. At 1200°, after one-half hour, the pearlyte has developed into the lamellar form, and there is little left of the granular ferrite. In Fig. 22 the ferrite has become entirely banded and striated.

A comparison of Figs. 13 and 17 shows a striking similarity in microstructure between the specimen held at 900° for one-half hour and that heated to 1000° and immediately cooled. It would be interesting to determine whether this point could be carried farther; that is, whether, for example, by prolonged heating at 800°, the microstructure of a specimen could be made to resemble that of a specimen immediately cooled from 1000°.

IV. Effect of Heat-Treatment on the Tensile Strength.

1. *Slow Cooling Immediately from T max.* — The original strength of the rolled bar does not seem to have been perceptibly affected by annealing until the critical point is reached. Here the curve drops considerably. This fall is well proven, as reference to the diagram will show that in the hollow of the curve at this point the results of all four observations are lower than either of the two next following. At about 750° the curve rises, reaching a maximum at 900°. From here it falls gradually to 1200°, and then rises again to 1300°, where our observations ended. This last point, though apparently irregular, seems to be well established, as the two results for 1300° are above both of those for 1200°.

Mr. Sauveur (in his paper of 1893) mentions a decided increase in the tensile strength of rail-steel at high temperatures. On this point Mr. Stead says, "The structure of the large grains" (the steel had been over-heated but not burnt) "consisted of bands or strips of ferrite and pearlyte arranged more or less parallel to each other. It would appear then that such a struc-

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Fig. 15. Section of bar heated to 900° C., and held at that temperature for three hours before cooling. 137 diameters.

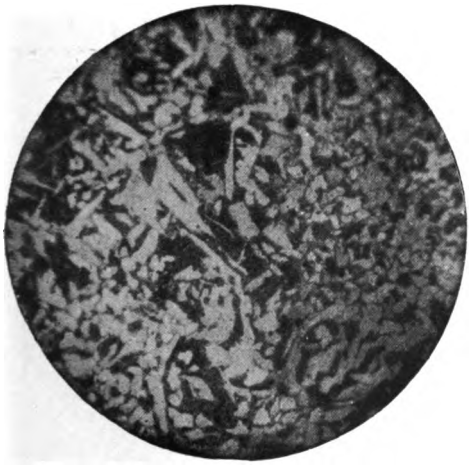

Fig. 17. Section of bar heated to 1000° C., and immediately allowed to cool.

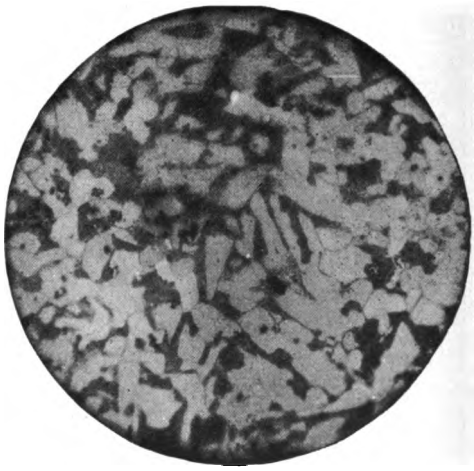

Fig. 18. Section of bar heated to 1090° C., and immediately allowed to cool. 137 diameters.

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ture, provided there is no inter-granular separation, is a remarkably tough one."

2. *Holding at T max. for Periods of Time.* — At 600°, prolonged heating lowers somewhat, but does not seem to affect substantially, the tensile strength. At 900°, the effect is not so marked as the appearance of the photographs would indicate. The tenacity of the bars held for one-half hour and for three hours (Figs. 13 and 15) is not greatly different from that of the bar (Fig. 12) which was immediately allowed to cool. The bar held for one hour (Fig. 14) was somewhat weaker. At 1200°, we find that the bar held for one-half hour (Fig. 20) is far weaker than any other specimen tested. The bars held for one hour and three hours (Figs. 21 and 22) are both stronger than the one which was immediately allowed to cool (Fig. 19).

At first it was thought that it would be possible to explain all the apparent irregularities in the curve of tensile strength by classifying the different appearances or size of the grain as representing different degrees of weakness or strength. This we are able to do in part, but not so satisfactorily as we would wish. A general relation seems to exist between the tenacity and the microstructure; namely, that the granular form is strongest with the smallest grain and weakest with the largest grain; and that the lamellar pearlyte is a very strong form of grain when well banded and pierced with ferrite. (Compare curves, Fig. 1.) This does not explain the well-marked fall in strength between 670° and 900°; nor are we able to find in the reports of any previous investigations any mention of this feature. Why the 1200° one-half hour bar (Fig. 20) is so very weak it is difficult to say. It bears a marked resemblance to the 900° one-hour bar (Fig. 14). The figures for the tenacity of the two resemble each other in being below the curve plotted. Judging from the figures plotted the 1200° one-hour and three-hour bars should have a grain resembling the 1300° bar more closely than does the 1200° one-half hour bar. The reverse seems to be the case. To be unable to make a better comparison between the appearance of the grain and the tensile strength is exceedingly discouraging. It shows that there is a new factor to be considered as affecting the microstructure, namely, the prolonged heating at a fixed temperature. Mr. Sauveur in his paper of 1893, after mentioning that there is a constant relation between the size of the grain and the proper-

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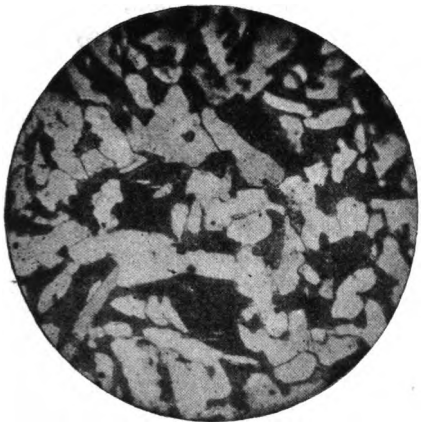

Fig. 19. Section of bar heated to 1200° C., and immediately allowed to cool. 137 diameters.

Fig. 20. Section of bar heated to 1200° C., and held at that temperature for half an hour before cooling. 137 diameters.

Fig. 21. Section of bar heated to 1200° C., and held at that temperature for one hour before cooling. 137 diameters.

Fig. 22. Section of bar heated to 1200° C., and held at that temperature for three hours before cooling. 137 diameters.

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ties of the metal says, "We must next endeavor to determine the extent to which the grain is affected by different finishing temperatures, and by each unit of the various impurities, carbon, silicon, sulphur, manganese, and phosphorus." The list is already a long one; and the ground which must be covered before the investigation of the effects of heat-treatment shall have been thoroughly accomplished is of vast area.

I have hesitated to draw many direct comparisons between our results and those recorded in previous investigations. In Mr. Stead's paper, as in many others, it is noticeable that the same

Fig. 23. Section of bar heated to 1300° C., and immediately allowed to cool. 137 diameters.

heat-treatment has different effects upon steels of different carbon-percentages; and, indeed, the effect of the other impurities, silicon, sulphur, etc., is hardly understood at all.

In the investigation reported by Mr. Sauveur in 1893, rail-steel was used, a comparison being made between the tenacity and the size of the grain. Some of Mr. Osmond's experiments (1893) were made upon medium steel (the exact carbon-percentage is not given). Mr. Stead experimented upon steels of different carbon-percentages; but in no instance did he use steel which was within 0.14 per cent carbon of that upon which our experiments were made. With our present lack of knowledge on the subject, it is impossible to say to what extent 0.14 per cent of carbon would affect the results of heat-treatment.

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Mr. Sauveur measured the grains in his experiments by means of a camera lucida and planimeter. Mr. Stead gives a table in which the diameter of the grains is compared with the maximum temperature of heating. His method of measuring is not mentioned.

The ground covered in this investigation was necessarily limited. Our experiments are too few and our knowledge of the subject is too limited to enable us to come to final conclusions. I therefore submit these results without further comment, hoping that at some time they may be corroborated by a more thorough investigation into the subject.

I here desire to thank Mr. Lilienberg for his kindness in furnishing us with the steel, also my fellow-student, Mr. C. H. Eckerson, who shared with me the laboratory-work upon which this paper is based.

I would acknowledge also our great indebtedness to Prof. Henry M. Howe, under whose direction and supervision the work was carried on.

SOME CAUSES OF EXCESSIVE HEATING IN BEARING METALS.*

By ROBERT JOB.

IT is a fact well known to those who have made a study of bearing metals that physical condition and structure exert a marked influence upon the efficiency of the metal in service. Formerly great stress was laid upon the chemical composition of the alloy, and comparatively little attention was paid to the effects of the different conditions of foundry practice, or to the relation between structure and efficiency. The natural results followed, and "hot-boxes" became prevalent in railway practice, especially so when weights and speeds became materially increased. Attention was thus directed to the production of cool-running and durable bearings.

As a result of carefully conducted service tests, the old copper-tin alloy of seven to one was found to be inferior as a

* *American Engineer and Railroad Journal*, February, 1900. The original paper is illustrated by some photo-micrographs which have not been reproduced here on account of their lack of clearness.

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bearing metal, and the copper-tin-lead composition was gradually introduced, at first combined with phosphorus, and later with this element present in very small amount, if at all, and then used only as a deoxidizing agent. The efficiency of a copper-tin-lead composition, other things being equal, was shown by Dr. Dudley to increase with the proportion of lead which was present, the amount being limited owing to inability to combine more than about fifteen per cent with copper to form a homogeneous composition. A large excess of lead was also avoided owing to the necessity of maintaining a strength sufficient to support the load, and also a fairly high melting-point in order to prevent fusion and running from the box if heating resulted.

During the past few years greatly increased attention has been paid to the microscopic study of the metals, and the importance of this method of investigation is becoming clearly recognized in view of the results which are being obtained through its use. In the course of an investigation to determine the alloy most efficient for general railroad use, we found it desirable to follow up this structure of bearing metals in order to note the influence of this as well as that of the chemical composition upon durability in service.

In order to secure information, a large number of bearings which had run hot and had been removed from cars of different railroads while passing over the Philadelphia & Reading Railway were taken for test. Fractures were made to show the general physical character of the composition, sections for microscopic examination were removed, polished, etched, magnified as far as necessary to show the structure to best advantage, and photographed. Analyses were also carried on at the same time, especially in cases where marked segregation of the metal was found to exist, in order to determine whether this result was due simply to an attempt at the foundry to form an alloy in proportions which were physically impracticable, or whether it was merely an effect of improper foundry manipulation. The marked crystallization which was often found in these bearings was also investigated in a similar manner. Also, in the majority of cases test sections were cut from the bearings, and the tensile strength and elongation determined, in order to find out whether in a given composition proper foundry practice would not be insured by placing a minimum limit upon the strength and ductility of the alloy.

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Side by side with these tests a considerable number of alloys have been prepared in the foundry to check the accuracy of the deductions, and to secure information as to the conditions of foundry practice necessary to give the greatest strength and ductility to the given composition.

By means of this study it has been possible to determine the causes of excessive heating in the large majority of the bearings examined, and we may summarize them as follows:

First, Segregation of the metals.

Second, Coarse crystalline structure.

Third, Dross or oxidation products, and an excessive amount of enclosed gas in the metal.

In addition to these, the lack of proper lubrication might be mentioned, though our investigation seems to show that a relatively small percentage of the bearings examined had been discarded owing solely to this cause.

Segregation has been found to be due in many cases to an attempt to alloy the metals in improper proportions, this being notably the case in some of the copper-tin-lead compositions in which an excessive proportion of lead had been introduced, resulting in the liquation not merely of a portion of the lead, but often also that of a part of the copper into "copper-spots," thereby producing surfaces of relatively high heating capacity, and ultimately causing "hot-boxes."

To a certain extent, these segregations may be prevented in a wrongly proportioned composition simply by a rapid chilling of the metal immediately after pouring, as for instance by the use of a cold iron mould. Such practice, however, is at the expense of the ductility of the metal, and causes a marked increase in brittleness with consequent rapid wear in service. High heating combined with rapid pouring and feeding is also a frequent cause of segregation, since under such conditions the metal in the mould remains for a considerable time in a molten condition, and by chilling gradually is given the greatest possible chance to solidify in definite natural alloys, throwing out whatever excess of metal may be present beyond these proportions, and thus resulting in segregation.

In actual service the effect of these segregations is readily understood, for it is evident that instead of an alloy of uniform hardness and heating capacity, there is a mixture, some portions

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of which are relatively very hard and others very soft, and this difference combined with that occasioned by the varying heating capacity of the different portions naturally localizes friction, and ultimately results in excessive heating. In a homogeneous alloy or composition no such conditions exist, and although, as is true of some compositions, some of the metals may be present, at least in part, in mere mechanical mixture and not as a definite alloy, yet the particles may be made so small by proper foundry practice that the friction throughout the bearing is practically uniform, and undue local heating is not liable to occur excepting through some outside agency.

The coarsely crystalline structure which was often seen in these defective bearings was in some cases found to be due to the composition of the alloy, antimony especially tending in this direction. In many cases, however, it has been traced to the foundry practice, often being due to rapid pouring at high temperature. Crystallization was also caused in some cases by the presence of an excess of various materials which were originally added as deoxidizing agents. Phosphorus and silicon are good examples. These, if added in suitable proportions, depending upon the condition of the metals, effect cleansing and free the metal from a large proportion of its enclosed gas, adding greatly to the fluidity, and thus rendering the casting less porous, and at the same time increasing strength and ductility often to a marked extent, correspondingly increasing the capacity for wear. Excess of these materials beyond the amount required for deoxidation appears not to be thrown off from the metal in the form of oxides very appreciably, but causes a crystallization which in a number of the bearings examined was so marked that it not only occasioned serious weakness and lack of ductility, but also such an increase in frictional qualities that cool running under the ordinary conditions of service was evidently an impossibility since the brasses had run hot and had been discarded from service shortly after the lead lining had been worn away.

One great advantage in the use of the microscope in connection with the deoxidation of these compositions lies in the fact that it becomes possible to tell quickly and with certainty the exact amount of the deoxidizer which is needed to combine with the oxygen present, without leaving more than a trace of the material behind in the finished casting to act as a weakener.

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The effects of this coarse crystallization upon the durability of the bearing are twofold. In the first place, increased local heating results in the same manner as in the case of segregated bearings, owing to the varying degrees of hardness and heating capacity of the constituents, and secondly, the ductility of the metal and the tensile strength are materially decreased. As the rapidity of wear with a given strength has been proved repeatedly by different experimenters to increase with the brittleness, it thus becomes evident that the durability of one of these crystallized bearings in service is bound to be defective owing to an excessive rate of wear, even though the heating which would naturally result should not occur.

Another very common source of difficulty found in defective bearings was the presence of particles of dross or oxidized metal mechanically enclosed, and also of large amounts of occluded gas in the metal. In the former case a hard cutting surface was presented to the journal, causing increased friction and hence heating. The presence of occluded gas in excess also tended in the same direction by reducing the actual bearing surface of the brass, and thus materially increasing the pressure. Such metal was naturally found to be very brittle, and to have worn rapidly in service. In the foundry practice, the presence of this enclosed matter is as injurious as in the bearings themselves, tending to cause sluggish pouring, unless the metal is heated to a very high temperature in which case crystallization and segregation — as shown above — are liable to result unless the speed of pouring is very carefully regulated.

The presence of dross enclosed in a bearing is simply a proof of carelessness in the foundry and is due either to defective skimming or to pouring from the bottom of the pot. In either case proper oversight will prevent the difficulty. An excess of enclosed gas, on the other hand, is ordinarily due to lack of proper deoxidation of the metals, though at times it is also caused by pouring at too low a temperature. Thus it indicates not necessarily carelessness, but rather a lack of knowledge upon the part of the foundryman of efficient foundry methods.

Turning now to the influence of the above-mentioned defects upon the tensile strength and elongation of the bearings examined, in every instance we have found the result which would be expected. The presence of dross or any foreign matter in the

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metal introduces an element of weakness, and thus reduces both the tensile strength and the elongation. Coarse crystallization produces the same result, the faces of the crystals forming the surface of least resistance, and thus facilitating fracture, and diminishing ductility. A test section taken from the bearing showed a tensile strength of only 10,500 pounds per square inch with an elongation of only four per cent in a 2-inch section. A bearing of the same composition if properly prepared in the foundry and free from crystallization would have a tensile strength of about 25,000 pounds per square inch and an elongation of about 13 per cent when the test sections were taken from the bearing in a similar manner.

In the porous brasses we naturally found the same lack of strength and ductility owing to the deficiency in the amount of the metal present in a given section. For example, a certain bearing showed a tensile strength of 15,000 pounds per square inch with an elongation of only six per cent; another showed a tensile strength of 18,700 pounds per square inch, with seven per cent elongation. Thus, we see that the influence of the various defects is clearly shown when metal of a known composition is subjected to the tensile tests, and it becomes possible to hold the foundry up to a high grade of excellence by means of these comparatively simple tests, with analytical and microscopic work as a basis.

Objection may perhaps be made that it appears rather arbitrary to place limits upon tensile strength and elongation in bearings, and that after all in practical service it is merely necessary to have, with a proper composition, a fairly strong homogeneous material, to obtain good results. In reply we will merely state that as a result of very carefully conducted service tests made by placing bearings of practically the same composition but differing widely in both tensile strength and elongation upon opposite ends of the same axles, we have invariably found that an increase of strength and ductility meant an increased life to the bearing in service and a lessening of wear, our results in this respect being in accordance with the deductions given by Dr. Dudley in 1892 before the Franklin Institute. As an instance of difference in efficiency due to these causes, we may cite a service test in which eight bearings each, of two copper-tin-lead compositions, were placed under tenders of fast passenger locomotives, one bearing

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of each kind being placed upon an end of each axle. All of the bearings were of practically the same composition, but the one set showed a tensile strength of about 16,500 pounds per square inch with an elongation of about six per cent, while the other had a strength of about 24,000 pounds per square inch with an elongation of about 13 per cent. This marked difference was due simply to the fact that in the one case the metal was porous, while the other was thoroughly deoxidized, and was close-grained and homogeneous. From time to time these bearings were removed and weighed, and the end-wear measured. As a final average result it was found that the more brittle set had worn thirty-five per cent more rapidly than the other set. The results of similar tests also have been in line with these results. Therefore it becomes evident that increased ductility and strength in the bearing of given composition means, as stated, an increased life for the bearings in service, and as this increased ductility necessitates also freedom from the defects which we have mentioned above, it is evident that the chances of cool-running are proportionately increased. These qualities are therefore not merely of theoretical interest, but have also an intensely practical value, and have a marked influence upon the success and economy of railway service.

Regarding the preparation of the sections for microscopic study, we have found it desirable to cut them from the center of the bearing, filing and polishing after the usual methods, and finally etching with an approximately deci-normal solution of iodin in potassium iodide — the time of etching being usually about one minute. This etching gives very satisfactory results in many cases, although in some cases etching with dilute nitric or with dilute chromic acid has shown the structure to better advantage. In this much depends upon the information desired. In ordinary work we have found that magnification to about thirty diameters is sufficient to show the general structure to good advantage.

In connection with our work it is clearly indicated that too much stress can hardly be laid upon the importance of the microscopic study of these alloys owing to the definite knowledge which is given regarding not only the composition of the alloy, but the general physical structure, the presence or absence of friction-producing agencies, and owing also to the check which is given over routine foundry practice.

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THE DILATATION OF IRON AND STEEL AT HIGH TEMPERATURE.*

By H. Le CHATELIER.

PROF. SVEDELIUS of the University of Upsala is the only one who has investigated with some precision the dilatation of steel at a high temperature,† and in his experiments the variations of temperature were so rapid that some doubts may exist concerning the accuracy of the figures obtained. With the collaboration of Mr. Chantepic, I have again taken up this investigation. We adopted in this inquiry the method of measurement used by Mr. Coupeau‡ in his researches on the dilatation of porcelain paste. A mirror of melted silica is so supported as to rotate around an horizontal axle in proportion to the difference between the dilatation of a support of Sèvres porcelain and the metal experimented upon; the mirror reflects a beam of light whose angular displacement is measured.

In the dilatation of iron and steel, three periods should be noted: the first corresponds to temperatures lower than the temperature at which the allotropic transformations begin; the second refers to temperatures higher than that of the end of these transformations, while between these two periods we have a third period corresponding to the range of temperature in which the allotropic transformations take place.

Dilatations Corresponding to Low Temperatures. — The iron experimented upon was a cast metal containing 0.057 per cent of carbon, 0.13 per cent of manganese, and 0.05 per cent of silicon.

The following table gives the mean values of a large number of experiments. The dilatations are expressed in millimeters, the initial length of the bar being 100 mm. The last line gives the true values of the coefficient of dilatation for each successive range of temperature of 100° C.

Temperature	0°	100°	200°	300°	400°	500°	600°	700°	800°
Dilatation		0.11	0.23	0.36	0.50	0.65	0.81	0.975	1.125
Coefficient $\times 10^6$	11	12	13	14	15	16	16.5	15	

* *Comptes Rendus*, August 7, 1899.

† A résumé of Professor Svedelius' experiments will be found in the present number of *The Metallographist.*

‡ *Bulletin Société d'Encouragement*, Oct. 1898.

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Experiments carried on with some true steels, i.e. with irons containing larger proportions of carbon, gave nearly identical figures.

The steels examined had the following compositions:

	1	2	3	4	5	6
Carbon, per cent	0.205	0.49	0.84	1.21	0.80	0.75
Manganese	0.15	0.24	0.24	0.24	0.15	0.15
Silicon	0.08	0.05	0.14	0.14	0.06	0.06

The following table gives the dilatations of these steels, as well as the dilatation of nearly carbonless iron as stated above.

Temperature	0°	100°	200°	300°	400°	500°	600°	700°
Iron		0.11	0.23	0.36	0.50	0.65	0.81	0.975
Steels, 1, 2, 3, 5 and 6		0.11	0.22	0.35	0.45	0.64	0.81	0.975
Steel 4		0.105	0.22	0.35	0.50	0.64	0.80	0.96

The differences between the dilatations of the various samples do not exceed 0.01 mm., which corresponds to the limit of precision of the experiments. It may therefore be inferred that iron and steel possess coefficients of dilatation nearly identical, whose value is 0.000011 at the ordinary temperature, and increasing regularly until at 750° C. its value is about 0.000017. The mean coefficient between 0° and 750° will then be 0.000014.

This identity of the coefficients of dilatation is in accordance with what we know concerning the constitution of steel, for this metal is made up of a matrix of iron through which are disseminated a small quantity of crystals of the carbide Fe_3C. At least four-fifths of the iron remains free, and it is natural that steel should very nearly retain the dilatation of pure iron.

Dilatations Corresponding to High Temperatures. — Above the molecular transformations, on the contrary, the dilatations of the various steels vary considerably according to their carbon contents, as indicated in the following table:

Carbon, per cent		0.05	0.20	0.80	1.20
Coefficient of dilatation	$\times 10^6$	15	17	22	29

This result agrees perfectly with our knowledge of the constitution of steel. Above the temperature of transformation, iron and the carbide Fe_3C constitute a true solid solution; in such cases, as I stated on another occasion,* there exists no necessary

* *Comptes Rendus*, June 12, 1899. *The Metallographist*, Vol. II (1899), page 334.

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relation between the dilatation of the mass and that of its constituents; the former may far exceed the latter.

Dilatations Corresponding to the Range of Transformation. — I have been unable to produce the transformations under absolutely reversible conditions. In this respect, therefore, my experiments are not more satisfactory than those of Prof. Svedelius. The following results are published tentatively only, it being my intention to again investigate this point:

Carbon per cent	0.05	0.20	0.50	0.80	1.21
Mean temperature of transformation	840°	768°	728°	730°	725°
Contraction in millimetres	0.26	0.23	0.21	0.08	0.10

The contraction is expressed in fractions of millimeter for a length of 100 mm.

These contractions are very irregular in successive experiments and are frequently followed by dilatations which nearly neutralized them. A great many of these anomalies could be explained on the ground that the change of dimensions is the resultant of two phenomena which may somewhat lag behind each other: 1° the molecular transformation of the iron, which is accompanied by a contraction of 0.26 mm., and 2° the dissolution of the carbide of iron in the new molecular condition of the iron, and which is accompanied by a dilatation amounting also to 0.26 mm. for carbon content of about 0.90 per cent.

THE MICROSTRUCTURE OF COKE AND CHARCOAL PIG IRONS.

By ALBERT SAUVEUR.

IN a recent paper entitled "Judging Pig Iron by its Microstructure"* Mr. K. Glinz contends that coke iron can readily be distinguished from charcoal iron under the microscope. The main difference between their microstructures consists according to the author in the shape of the particles of graphite, which in the case of coke iron are present in long fibers, while in charcoal iron the graphite occurs in much shorter and irregular streaks, frequently assuming a star-like appearance.

A greater irregularity of structure is, according to the au-

* *Stahl und Eisen*, Nov. 15, 1899, page 1061.

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thor, characteristic of charcoal iron and may, in his opinion, account in part for its superiority over the more regular, but, possibly on that account, weaker coke iron.

The paper is accompanied by six photomicrographs, which, however, are not sufficiently clear to warrant their reproduction in these columns.

Mr. E. Heyn* takes exception to Mr. Glinz's conclusions, arguing that there cannot be any essential difference in microstructure between coke iron and charcoal iron of the *same composition and cast under exactly the same condition,* since the microstructure of a cast metal is solely a function of the chemical composition, the casting temperature and the rate of cooling. A difference of treatment *in the furnace* cannot as such influence the microstructure proper. Only through the action it has upon the chemical composition can it alter the structure.

Mr. Glinz must show us that two samples respectively of coke and of charcoal iron having the same composition and cast exactly under the same conditions differ structurally in the way he describes before we can accept his conclusions.

Mr. Heyn argues rightly that such differences as the author describes must be due, if the samples have the same, or nearly the same, composition, to a difference in the casting temperature and in the rate of cooling. With this reasoning the writer is in full accord, and as additional support to Mr. Heyn's view he offers the two photomicrographs appended to this note.

Both show the structure of the same coke iron, representing two tests taken from the same cast of a blast furnace and at a very short interval of time. The iron contains 1.54 per cent silicon and 0.075 per cent sulphur. The sections prepared for microscopical examination were carefully polished but not etched, as the purpose was to reveal the shape of the graphite particles, and this can be more clearly seen in the samples before etching. The magnification is in both cases 56 diameters.

The sample represented by Fig. 1 was cast in sand, while Fig. 2 represents the center of a chilled bar of exactly the same dimensions. In Fig. 1 we have the structure which Mr. Glinz says belongs to coke iron, characterized by long, often very thin plates of graphite, and in Fig. 2 we see the structure which he

* *Stahl und Eisen*, Jan. 1, 1900, page 36.

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considers typical of charcoal iron: short, intricate particles of graphite, showing a tendency to assume a star-like appearance.

This would seem to be conclusive proof that the two types of structure described by Mr. Glinz are simply due, as argued by Mr. Heyn, to differences in casting temperature and in the subsequent rate of cooling.

Charcoal iron is generally cast much colder than coke iron, and to this fact must be attributed the difference of structure

Fig. 1. Coke Pig Iron cast in sand. Magnified 56 diameters.

Fig. 2. Same Pig Iron as in Fig. 1. Center of chilled bar. Magnified 56 diameters.

recorded, seeing that the structure considered characteristic of charcoal iron can so readily be imparted to coke iron by hastening its rate of cooling.

Mr. Glinz advocates the use of galvanic treatment to develop the structure of cast iron. By lightly nickel-plating polished sections he finds that the various constituents are sharply brought out. Judging from his photographs, however, it would seem that this can be accomplished more perfectly by proper polishing followed or not by etching or by the careful heating of the sample.

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The Effect of Annealing on the Magnetic Properties of Steel Sheets.—The following is a condensed translation by Dr. Richard Moldenke (*Iron Trade Review,* February 15, 1900) of a paper by Mr. Hans Kamps (*Stahl und Eisen,* December 1 and 15, 1899).

The behavior of the laminated field magnets and armature cores in dynamo construction is so peculiar at times that much attention has been given to this matter by the great electrical companies. Methods with tedious details have been devised, the object of which is to produce a uniformly reliable product so far as its magnetic permeability is concerned. The need of more light on the subject is in fact so urgent that a special committee was authorized, at the recent convention of the American section of the International Society for Testing Materials, to look into the behavior of iron and steel under electrical influences. Mr. Hans Kamps gives us some data of interest on one phase of the question. The problem of the chemical composition of iron as related to its magnetic properties is still unsolved. We know that the three great divisons of iron give corresponding readily distinguishable magnetic effects. When, however, it comes to a study of each division, that is, wrought iron, steel and cast iron, the laws of magnetic permeability, which should be well defined in their relation to the variations in composition, cannot be traced as yet. The reason for this is patent. Not only does the chemical composition affect the magnetic properties of iron, but also the heat and mechanical treatment it has received. As long as it is impossible to give two pieces of sheet iron exactly the same treatment in the fire it is useless to analyze them and predict their magnetic permeability. It is necessary, therefore, to learn the actual effects of heating and forging a piece of iron or steel in order that a rational system of annealing dynamo material may be devised. It is a well known fact that annealing sheet iron for magnetic purposes sometimes improves it, and sometimes does the reverse. Why this should be the case we cannot as yet say.

The points to be investigated would seem to be: (1) the maximum temperature of the anneal; (2) the duration of the heating process, and (3), the rapidity of heating and cooling, with a noting of the effects of possible irregularities in process. Mr. Kamps suggests the use of a small electric furnace to carry out these experiments, as it is impracticable to do work of this

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kind in regular factories without serious interference with the daily routine. The experiments made on the duration of anneal given by the author show that the results were negative, no effect due to a variation in time of anneal being proven. Nevertheless the data are of great interest in connection with the study of the theories of magnetic changes during annealing. A series of eight test strips taken from two sheets was secured. These were all heated to the same temperature, but varied in length of anneal from 35 hours for the short anneal to 48 hours for the long. The Koepsel system of testing was used, and before the separate trials were made each piece was magnetized and demagnetized several times. The influence of the earth's magnetism was carefully eliminated and the mean of a number of measurements taken. The maximum difference in losses by hysteresis in the eight samples before annealing amounted to 21.6 per cent of the highest amount. Four of the samples were now subjected to an annealing process of short duration. While all of the samples were improved magnetically, yet the maximum difference in the losses by hysteresis increased to 56.5 per cent of the highest amount as compared with 16.8 per cent before annealing these particular four samples. The second lot of four test strips were now annealed for a long period. The results showed no variation of note when compared with those from the short anneal. The second lot was now re-annealed for a short period and the first lot divided, one portion being annealed for a short period again and the other for a long one. The results show a marked deterioration in the magnetic qualities. All samples were now subjected to a further long continued anneal. Here again the result was a much poorer magnetic quality. The summary of the tests would seem to indicate that the first anneal of the samples made a material of much better magnetic qualities, and all succeeding trials were useless, in fact detrimental.

The questions which now arise are: (1) In what manner does the anneal affect the magnetic character of the sample? (2) Why does it improve the material in the one case and spoil it in the others? (3) Why is the annealing effect irregular in the various samples? We have as yet no satisfactory solutions to record. Light is shed upon the subject, however, by the recent discoveries on the nature of hardening steel and the microstructure of metals. It is to be supposed that magnetic changes due

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to annealing must be accompanied with physical or chemical changes in constitution, such as a change in the grain, the microstructure, and the form of carbon and iron. A continuation of these influences must be of far-reaching effect upon the magnetic properties. For instance, we know that a hardening of the material means an increase in the loss through hysteresis. Again the fact is patent that hardening a steel means that its carbon has been changed in form. Heat a piece of ingot iron or soft steel to cherry red and the carbon is in one form only — hardening carbon — and is combined with the iron as "Martensite." Cool this iron to 1560° F. and pure iron begins to separate out. The interesting fact may now be reported that at this critical point the temperature coefficient for the electrical resistance is suddenly modified. Above 1560° F. there is no change noticeable in this coefficient. Cool the iron down to 1,380° F. and another critical point is reached, evidently as Mr. Kamp thinks, the one at which the nature of iron changes from a hard to a soft one. This point is the upper limit of the susceptibility of iron to magnetization; it will neither retain residual magnetism nor receive any by induction. The third and last critical point is reached when cooling down to 1250° F. — the point of recalescence — when the hardening carbon changes to carbide carbon. Below this point it is impossible to harden steel, and this naturally affects the magnetic properties likewise. These critical points are reversed when the iron is heated. The hardening carbon may be readily separated from the carbide carbon by chemical means the former being soluble only in hot acids, while the latter escapes as a gaseous carbo-hydrate when treated with cold nitric or hydrochloric acids.

The much mooted question whether iron is allotropic or not is founded upon physical phenomena. The circumstance that the various critical points of steel come closer together, in fact unite into a single one, when a high carbon steel is reached, indicates that several explanations of the hardening property shown by steel are possible and of equal weight. Osmond and his followers attribute it to the presence of allotropic hard iron, which is prevented from becoming soft by the presence of carbon. Ledebur, on the contrary, together with his adherents, can get along without allotropic iron and attribute the hardening of steel to the amount of hardening carbon present alone. Howe

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takes a position half way between, with the carbo-allotropic theory, by which the combination of hardening carbon and allotropic iron are said to account for the peculiar power of steel to harden when heated to a high temperature and quenched. If we were to assume that a greater or smaller amount of allotropic hard iron in the sheets to be magnetized accounts for the greater or smaller losses by hysteresis, the influence of hardening carbon is not so apparent, for the very softest steel is universally used for laminated magnets. As this amount of carbon is seldom above a few hundredths of one per cent, and the proportion of hardening carbon in this total carbon reduces this still more, we can assume that a well annealed piece of dynamo sheet steel contains only traces of hardening carbon, or not enough to account for marked changes in its magnetic properties. A more potent phenomenon is the remarkable coincidence of the critical points at which the forms of carbon change and the magnetic permeability is lost. The change of the magnetic properties with the change from soft to hard iron may be best attributed to the last mentioned occurrence. It is impossible to explain the increased losses from hysteresis by cold working in any other way than a change in iron from soft to hard. Cold working by damaging the crystalline structure, creating a greater number of smaller crystals, may increase the adhesion of the crystalline mass and therefore make the work of remagnetizing a little heavier, but then a change in the form of carbon is not brought about by mechanical means.

There seems to be only the allotropic theory of iron remaining to account for the changes in the magnetic properties of an iron under anneal, for, take the well known theory of fatigue of metals and apply it to the "ageing" of dynamo sheet iron, and one finds that the carbon conditions here are not changed at all. Nevertheless the continued remagnetization must necessarily affect the molecular conditions somewhat by reason of the internal friction produced, and when the carbon is very high its effect more than counterbalances that of a possible change from soft to hard allotropic iron, for in making permanent magnets the effect of the carbon present is especially intensified by the addition of chromium, tungsten, titanium, etc. Summarizing the above points we find that the magnetic changes produced by annealing are caused by the changes in the proportion of hard and

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soft iron present as well as hardening and carbide carbon, but what their relative value may be must be left an open question. In order to make use of the above explanation for obtaining practical results we must revert to the phenomena observed in the rapidity of the cooling of an iron at the critical points, this factor determining the proportion of the unchanged allotropic iron and hardening carbon remaining behind. It would appear that the change from soft to allotropic hard iron, as well as carbide to hardening carbon can be effected instantaneously, whereas the reverse operation is a matter of some time. It would seem to follow that the duration of the cooling process in annealing sheet iron has everything to do with the magnetic changes effected therein, while it is immaterial whether the metal was brought to the required temperature quickly or not. If a very hot piece of sheet iron is cooled fairly quick the temperature drops rapidly about the critical points and the consequent hardening noticed has a very bad effect upon the magnetic properties observed. Rolling sheet iron produces this effect at every pass, and careful annealing is always resorted to if good magnetic results are required. It is sometimes noticed that the same anneal improves some plates and spoils others. This is due to the different position of the critical points in the two materials, a fluctation in the speed of cooling striking one point slowly with good results, and the other rapidly with poor ones. It is therefore necessary to heat up to a point beyond the highest critical point of the material and then cool slowly and uniformly. The presence of manganese and nickel reduces the critical points very materially. Thus a 12 per cent manganese steel would be expected to be inert to magnetism at ordinary temperatures. This is really the case with Mr. Hatfield's manganese steel.

Regarding the influence of chemical composition upon the magnetic properties of an iron much can be said. So far only iron and carbon have been found of direct influence, the other elements being but of secondary importance. Thus silicon and aluminum remove gases and thus increase the permeability of an iron. Phosphorus, manganese and silicon affect the size of the crystal and thus also the magnetic properties. The effect of varying quantities often gives opposite results. Thus a 12 per cent manganese steel cannot be magnetized, but add more manganese and it is again amenable to magnetic influences. The size of

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the grain is quite influential, for with it the adhesive forces change, and the molecular friction incident to the demagnetization and remagnetization varies likewise. One can readily see a relation between size of grain and magnetic permeability. Unfortunately we can prove little of the deductions given above by direct experiment, for we cannot distinguish the allotropic states of iron by chemical analysis, though they are theoretically in existence. Nevertheless systematic trials should be made right along to get at a basis for properly annealing sheet iron for dynamo purposes so that the highest magnetic results may always be obtainable.

Measure of the Abnormal Variations of Length and Temperature Taking Place in Iron and Steel During the Recalescence.* — Professor Svedelius* states that if a wire of copper and a wire of iron or steel are heated and cooled side by side, it is found that the copper expands and then contracts uniformly, while the iron or steel wire exhibits an abnormal contraction on heating and an abnormal dilatation on cooling when the temperature of the thermal critical point (the recalescence) is reached. He finds that the contraction during heating is notably less than the dilatation during cooling and that the contraction begins at the same temperature in iron and soft steel, but covers a range of temperature which is the greater, the less carbon present. The dilatation takes place at a higher temperature and is of shorter duration in iron than in steel. Both the contraction and the dilatation are of a greater magnitude in soft steel than in iron or hard steel. The variations of length increase with the carbon content, and are maximum when the metal contains 0.60 per cent of carbon; as the carbon content further increases from 0.60 to 1 per cent, the variations of length decrease. In the case of iron and of soft steel both the contraction and the dilatation appear to take place in two stages which are confounded in harder steel. The position of these critical variations of length corresponds with the thermal critical points. The variations of length, however, are greater in the case of soft steel than in iron and hard

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* *Philosophical Magazine*, August, 1898. See also "The Dilatation of Iron and Steel at High Temperatures" by H. Le Chatelier in the present number of *The Metallographist.*

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steel, while the thermal critical points reached their maximum in hard steel, and decrease in magnitude with the carbon content.

The following table gives the values of the dilatations and contractions in the case of steel wires containing various amounts of carbon:

Carbon per cent	Dilatations during heating		Contractions during cooling	Coefficient of dilatations
	from 0 to 600°	from 600 to 800°	from 800 to 600°	from 0 to 800°
0.9	0.00750	0.00340	0.00335	0.0000135
0.6	0.00770	0.00245	0.00220	0.0000125
0.3	0.00760	0.00105	0.00105	0.0000110

It is seen that the dilatations taking place between 600° and 800° C. and the contractions between 800° and 600° diminish with the carbon content, while the dilatation between 0° and 600° is quite independent of it.

The Strains in Completely Hardened Steel.* — O. Thallner observes that Reiser was the first to point out that the strains produced in steel by hardening and deducible from the changes in volume of the metal, are the cause of the hardening, and Barus and Strouhal proved that in hardened steel considerable differences both in volume and specific gravity existed between the strongly hardened outer layers and the less strongly hardened inner layers, and that the strains resulting were the causes of cracks in hardening.

The author made a number of experiments for the purpose of ascertaining the influence exerted by manganese and by silicon and the changes in volume during hardening, and these led the author to doubt the accuracy of the existence of pressure strains.

Two kinds of strains must exist: (1) those which occur in steel of small cross-section that has been evenly hardened throughout, and (2) those which occur in steel of larger cross-section, due to the unequal changes in volume of the surface and the interior.

The first of these also occur in steels of large cross-section and to the greatest extent on the surface. If steel of small cross-section is so hardened that it becomes of the same degree of hard-

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* *Stahl und Eisen*, Oct. 15, 1898. Reviewed in *Journal of the Iron and Steel Institute*, 1899, Vol. I.

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ness everywhere, it may still crack, although this cannot be considered as due to pressure exerted by the hardened and contracted outside on the interior.

If steel of large cross-section is hardened, cracks may commence at the surface as well as within the metal. It is not possible, however, for such cracks to form within the metal if they are due to pressure exerted by the outer hardened skin on the softer core.

So, in accounting for such strains, the author first passes in review the present theories of the hardening process, and, drawing attention to the changes in texture and volume which take place, considers that the strains referred to have their origin in these causes.

In a later paper O. Thallner* considers the question of the strains that occur in hardened steel of large cross-section.

These are due, the author observes, to the changes in volume and in shape that take place during the hardening. He discusses these seriatim, and observes that numerous experiments made at the Bismarck Works have shown that steels of different chemical composition may be divided into two main groups as regards their behavior during the hardening process: (1) those which become shorter, and (2) those which become either shorter or longer. These two groups are not separated from each other by any definite line of demarcation.

It is dependent on the chemical composition, and in the case of pure carbon steels, lies at about a percentage of 0.90 carbon. In the case of steels of the first group and those of the second, which become shorter when hardened, an increase in the thickness and width is always observed.

The larger cooling faces always assume a concave form. In the case of steel belonging to the second group, which becomes longer when hardened, an increase in the dimensions both as to width and thickness may occasionally be observed, but the larger cooling faces are never concave. They are, indeed, usually slightly convex.

A series of results are given of measurements made in connection with five samples of crucible steel and eight of basic open hearth steel. The former became shorter and the latter

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* *Stahl und Eisen*, April 1, 1899.

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longer after hardening. The various kinds of strain which can result are then enumerated and considered.

Judging Pig Iron by its Microstructure.— Mr. K. Glinz publishes in *Stahl und Eisen* for November 15, 1899, a paper in which he describes the difference between the microstructure of coke and of charcoal pig iron. His remarks are illustrated by six photomicrographs. He also describes and advocates galvanic treatment for the development of the microstructure of cast iron.

Mr. Heyn in *Stahl und Eisen* for January 1, 1900, takes exceptions to Mr. Glinz's conclusions. For the substance of these papers, the reader is referred to a note on "The Microstructure of Coke and Charcoal Pig Irons" by Albert Sauveur, in the present number of *The Metallographist.*

Some Modern Theories on the Cause of Hardening in Steel.* — Messrs. F. W. Harbord and T. Twynam review at length and very ably the current theories of the hardening of steel. The subject, however, has been fully discussed elsewhere in these columns. The authors conclude their paper by the following remarks:

For convenience, the allotropic theory may be divided into two parts: (*a*) Does an allotropic form of iron exist? (*b*) Assuming its existence, is it the cause of hardening of steel on quenching? Let us first consider the evidence in favor of an allotropic form or *Beta*-iron:

(1) It is admitted, even by the opponents of the allotropic theory, that the critical points Ar_2, Ar_3 exist in practically carbonless iron (electrolytic iron), and this in the widest sense of the word allotropic, as defined by Professor Roberts-Austen, is the very strongest evidence of such a change occurring.

(2) That each critical point is accompanied by a marked change in the microscopic structure of the metal, and no such changes occur except at the critical points.

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* *Journal of the West of Scotland Iron and Steel Institute*, March 1898.

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(3) That the point Ar_2 corresponds almost exactly with the disappearance of the magnetic properties in soft iron.

(4) The extraordinary difference between the ferromagnetic properties of iron at 100° and at just above Ar_3. Amongst others that the electrical resistance is ten times greater at this point than at ordinary temperatures, and that it does not *perceptibly increase above* Ar_3.

On the other hand, we have:

(1) The work of Arnold, from the results of which he maintains that the critical point Ar_3 is due to dissociation of the sub-carbide $Fe_{24}C$, while the point Ar_2 marks the passage of iron from a plastic to a crystalline state.

(2) That etching at a red heat with calcium chloride gives no indication of an allotropic modification of iron.

In our opinion the evidence in favor of an allotropic modification of iron (confining ourselves to Roberts-Austen's definition of the word) is so strong as to amount to a practical certainty, the fact alone that evolutions of heat take place at Ar_3 in carbonless iron being fatal to Arnold's contention that it can be due to a dissociation of a subcarbide. It must also be remembered that iron with about 0.9 per cent carbon is a eutectic alloy — that it is a physical and not a chemical compound. With regard to Arnold's suggestion that Ar_2 marks the passage from the plastic to the crystalline state, it appears to us that this change, assuming it to take place, is covered by Professor Roberts-Austen's definition of allotropy. The interesting, but purely negative results of Saniter on etching at a red heat in face of the other evidence above cited of the extraordinary changes in structure and properties of the metal at and about the critical points, cannot carry much weight as evidence against the existence of *Beta*-iron.

Turning now to the evidence for and against the theory that the hardening of steel on quenching is due to presence of *Beta*-iron, we have:

(1) The fact that it has been clearly proved that iron practically free from carbon undergoes considerable hardening on quenching.

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(2) Such hardening, according to some experimenters, bears distinct relation to the critical points; according to others, it is simply a function of the quenching temperature.

(3) That martensite, of which high carbon steels on quenching above the critical point almost entirely consist, is present to a considerable extent (77 per cent) in steels almost free from carbon when suddenly quenched. Such martensite, however, contains much less carbon, and is much softer than that present in high carbon steels.

(4) Manganese steel containing about 12 per cent of manganese does not show any marked retardation during slow cooling. It is hard and non-magnetic.

With regard to this part of the allotropic theory — that the hardness of quenched steel is due to *Beta*-iron retained as such by sudden quenching — the evidence cannot be regarded as so conclusive as that for the existence of *Beta*-iron. The fact that carbonless iron is capable of hardening on sudden cooling is a distinct point in favor of the contention, and the evidence for and against such hardening bearing a distinct relation to the critical points has been dealt with in the paper. The large proportion of martensite present in all quenched steels, whatever the content of carbon, may be used as an argument both for and against allotropy, but it appears to us to rather support the view that hardening is due to a change in the iron itself, and that such change is intensified by the presence of carbon.

The exact part, however, that allotropy plays in the hardening of steel still remains a question, and the allotropists need the solution of some incidental physical problems before they can prove their case. It will, nevertheless, be admitted that they have at least done this — they have given a rational classification of the action of foreign elements, connecting the carbonised steels to the manganese and nickel steels; they have shown that steel above the critical point is a solution. These results are no small gain to our knowledge of the physics of iron, and the position taken by allotropists in relation to various hypotheses advanced cannot be better expressed than in the words of Mr. Osmond: "They see no reason why their views should not be reconciled with the dissolved carbide of Mr. Sauveur, with the carbide of

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iron of Mr. Howe, the double carbides of iron and manganese of Mr. Hadfield, and the molecular stresses of Mr. André le Chatelier."

The Heat-Treatment and Microstructure of Steel.* — Mr. A. Campion says that at the present time the chief use of the microscope in the examination of steel is as a detective agent, more especially in determining the form of heat treatment to which the steel has been subjected, and it is in this direction that the author considers the greatest amount of assistance may be expected from the microscope. It is generally possible to determine whether a steel has been annealed, oil-tempered, etc., and if these operations have been properly carried out. It is also possible to determine whether the steel has been finished at a normal temperature, too high, or too low a temperature; and in some cases a very fair idea can be obtained as to the temperature to which the steel had been heated before quenching, and that at which it was quenched. The author would recommend this subject to practical men as one of intense interest, which can be investigated by the aid of very simple apparatus and low powers of the microscope, and which presents a very wide field for research.

The constituents of steel, ferrite, pearlyte, cementite and martensite are described at length, and the constituents sorbite, austenite and troostite briefly mentioned.

Six samples of steel containing from 0.06 to 1.21 per cent of carbon were subjected to various heat treatments and their physical properties and microstructure ascertained.

Some experiments were conducted to ascertain the effect of annealing at various temperatures and for different lengths of time upon the physical properties of two samples of steel containing respectively 0.18 and 0.36 per cent of carbon.

These experiments and the results obtained are described below:†

A number of one and a-half inch round bars of the same steel were obtained and treated in various ways as described below. The first set treated had the following composition:

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* *Journal of the West of Scotland Iron and Steel Institute*, 1900.

† See also a paper on "The Relations Between the Structure of Steel and Its Thermal or Mechanical Treatment," by A. Sauveur in *The Metallographist*, Vol. II (1899), page 264.

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	Per cent.
Carbon	.180
Silicon	.055
Sulphur	.055
Phosphorus	.071
Manganese.	.500

The bars after treatment were turned up to three-quarters of an inch, mechanical tests made, and micro-sections prepared.

No. 1 (Table VII) was heated rapidly to 820° C. and maintained at that temperature for twenty minutes, then slowly cooled by covering with lime.

The microstructure is fine grained: under a high power the pearlyte is finely striated, and the ferrite in small meshes evenly distributed.

No. 2. Heated as in No. 1, but maintained at the temperature for 1¾ hour. The structure is coarser than No. 1, the ferrite grains being larger, and not so uniform, and increasing in size towards the centre.

No. 3. Heated in closed box with lime for 36 hours in flue of billet reheating furnace, and allowed to cool in the furnace during 32 hours at week end. Structure is very coarse, large grains of ferrite.

TABLE VII.

Results of Mechanical Tests of Soft Steel Annealed in Various Ways.

No.	Size of sample	Breaking strain, tons per sq. in.	Elongation per cent 3 in.	Elastic limit tons per sq. in.	Cont. of area per cent.
1	.798 dia.	29.1	38.3	14.7	58.3
2	"	28.6	39.1	14.8	62.3
3	"	27.2	36.0	14.2	57.6
4	"	28.1	35.7	14.5	56.3
5	"	28.0	39.3	14.9	59.9
6	"	29.3	35.8	15.3	56.3
7	"	28.6	41.9	14.8	61.6
8	"	27.6	35.0	14.2	55.1
9	"	29.0	39.2	15.1	60.0
10	"	28.8	36.0	14.8	59.2
11	"	29.0	38.0	15.2	61.0
12 N	"	29.8	33.3	15.4	57.6
13	"	26.7	38.3	14.2	61.3
14	"	27.3	33.8	14.2	53.4

No. 4. Heated to 1,000° C. for fifteen minutes, and covered with sand to cool slowly. Structure coarser than Nos. 1 and 2, but not so coarse as No. 3.

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No. 5. Heated to 750° C. for 1 hour, and covered with sand to cool. Structure very fine, ferrite in very small meshes. Pearlyte and ferrite very intimately mixed and distributed throughout the section. This and No. 11 are the finest grains in the series.

No. 6. Heated very rapidly to 840° C. for half-an-hour, and allowed to cool in closed furnace, ferrite in small meshes, but grain not so fine as No. 5.

No. 7. Heated two hours at 750° C., cooled in furnace till below Ar, and again heated for two hours at 750° C., and slowly cooled. The grain structure is not quite as fine as Nos. 5 and 6. The heating was not quite as rapid as in those cases which may account for it; or on the other hand, the heating may have been too prolonged; at any rate the double heating appears to have no advantage.

No. 8. Heated in closed box with ore for forty-eight hours in flue of billet heating furnace, and allowed to cool in furnace during thirty-one hours. Structure very coarse.

No. 9. Heated to 930° C. for two hours, and slowly cooled, coarse and uneven structure. Ferrite grains fairly large and in patches.

No. 10. Heated very slowly to 850° C., and slowly cooled. Structure rather coarse, and somewhat resembles that of No. 4.

No. 11. Heated to 740° C. to 760° C. for half-an-hour, and covered with ashes. Structure almost identical with that of No. 5.

No. 12. Steel as rolled, fairly coarse structure, pearlyte present as granular variety.

No. 13. Heated from 630° C. to 670° C. for three hours, prolonged heating has started crystallization.

No. 14. Heated to 1160° C. for a few minutes, and covered with ashes, coarse crystalline structure.

Six bars of the same size as before, but having a higher carbon content, were treated at various temperatures.

The steel had the following composition:

Carbon	.360
Silicon	.075
Sulphur	.038
Phosphorus	.039
Manganese	.752

The results of mechanical tests are shown on Table VIII.

No. 21. As rolled coarse structure ferrite in large meshes.

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TABLE VIII.

Results of Mechanical Tests of Steel Containing 0.36 per cent Carbon Annealed in Various Ways.

No.	Size of sample	Breaking strain, tons per sq. in.	Elongation per cent 3 in.	Elastic limit tons per sq. in.	Cont. of area per cent.
21	.798 dia.	41.8	23.8	21.6	51
22	"	39.9	25.3	21.0	43.5
23	"	42.0	29.1	21.4	49
24	"	42.0	25.0	20.4	40
25	"	37.4	26.6	19.6	60
26	"	39.6	26.6	20.2	49

No. 22. Heated rapidly to 810° C. for fifteen minutes and cooled in ashes. Fine structure ferrite in small meshes.

No. 23. Heated rapidly to 760° C. for twenty minutes, covered with ashes to cool slowly. Structure rather finer than No. 22.

No. 24. Heated to 1100° C. for a few minutes and cooled slowly. Structure crystalline, but not as much as No. 21.

No. 25. Heated to 680° C. — 710° C. for thirty-five minutes — and covered with sand. This structure is very fine (the finest of series), the ferrite in exceedingly small meshes. It will also be observed that this sample has best mechanical result. Under high power the pearlyte is exceedingly finely striated.

No. 26. Heated to 980° C. for ten minutes, and cooled in sand. Structure shows signs of crystallization, and is intermediate between Nos. 22 and 24.

From a consideration of the above and of a number of similar experiments it would appear that in the case of soft steel, such as is usually supplied for constructional purposes, there is a fairly wide latitude of temperature to which the steel can be heated to anneal it or at any rate considerably increase its ductility. The best results, both as regards mechanical results and the microstructure, are given by Nos. 5, 7, and 11 cases, in which the heating was fairly rapid to the required temperature, which did not exceed 750° C. This is quite in accordance with the results of Brinell, i.e., to obtain fine grain the steel must not be heated above the point which he denoted by W (about 730° C. red heat). The change from hardening carbon to cement carbon takes place at some 40° or 50° below W and denoted by V on the scale of Brinell, but now more generally designated Ar_1. The time element, however, plays a very important part in the reaction, as

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pointed out in an earlier part of the paper. It is therefore necessary to heat to the point W, or Ar_1, to ensure the completeness of the change during cooling, which should be very slow through V. The same transformation of carbon may also be completely brought about by a prolonged heating at the temperature V, as is shown in the case of No. 13, which was heated for three hours between 630° C. and 670° C.; but the steel under these conditions does not possess the finest grain possible — a quality universally sought after — but is very liable to develop a crystalline structure, in fact, this is, more correctly speaking, a case of soaking and not annealing.

In the case of the second series of tests and structures given of the higher carbon steel it appears that it is necessary to keep the temperature within more narrow limits, and a crystalline and coarse structure is more easily produced than in the softer steel. The results, as far as they go, are also in keeping with those of Brinell.

With regard to the question of defining annealing, this is not altogether a simple matter; but probably that given by our late president, Mr. Paul, in his opening address in October, 1897, is as good as any yet put forward. Mr. Paul said — "Annealed steels should possess the highest possible ductility with lowest possible tensile for that particular grade, accompanied in all cases with fine grain. Such steels would then be in the most suitable condition to withstand dynamic tests such as the percussive blow of a shot, or the drop test of a weight falling from a height of 10 to 30 ft."

Before any definite conclusion can be drawn or definite statements made as to the best methods of annealing any particular grade of steel, a large number of points must be taken into consideration, such, for instance, as the mass of material operated upon, and the amount and description of work to which it has previously been subjected — matters too often left out of consideration. The material experimented upon must also be tested dynamically, as that method shows far better the qualities of the steel than the ordinary tests for tensile strain and elongation.

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Rail Steel: Its Chemistry and Heat-Treatment.* — Mr. W. R. Webster calls attention to the importance of finishing steel rails at the proper temperature.

In rails, the chemical composition and heat-treatment are of equal importance, and we must not overlook one in considering the effects of the other; yet this is done, and cases cited where good rails chemically have given poor results, and where rails of inferior chemical composition have given good results; also where rails of low carbon steel give better wear than those of higher carbon steel. These differences might have been, and probably were, produced by differences in the heat-treatment of the rails, and the conflicting results are not a sound argument against using chemical composition as a guide. The problem must be considered as a whole, that is, the composition of the steel, its manufacture, heat-treatment, and the section of the rail.

In all rails there is a large mass of metal in the head that holds the heat much longer than the thin metal in the flanges. This at once shows the great difficulty in finishing the head at a low enough temperature, as the light flange cools too quickly, and you cannot after this continue the work of rolling on the head down to a sufficiently low temperature.

In the heaviest sections of rails this difficulty is greatly increased as the large head holds the heat much longer than in the lighter sections. Each point of carbon added in these heavy rails makes it still more difficult to produce a fine structure in the head of the rail, as a finishing temperature that would give satisfactory results with the medium carbon heavy rail might be too high for the higher carbon steel and give a coarse structure.

The beneficial effect of finishing a heavy rail at the proper temperature in rolling is shown in re-rolling heavy rails that have not given good service in use, but after this second rolling they have given satisfactory results. This is accounted for in the first place by the annealing action of the furnace in heating the rails up to a low heat for rolling, the coarse grain being removed in this way, and they are not heated up high enough to form it again. Then the final work of rolling on the head is at a low temperature, as the flange is in a condition to allow this work at proper temperature.

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* *The Railroad Gazette*, Feb. 16, 1900, page 99.

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METALLOGRAPHIC NOTES.

Phosphides and Sulphides under the Microscope. — In a recent lecture before the Cleveland Institution of Engineers, at Middlesborough, England, Mr. J. E. Stead demonstrated that the phosphides and sulphides present in cast iron and steel could readily be detected under the microscope, after suitable preparation of the samples.

The report of this most important discovery of Mr. Stead is fully confirmed by some private communication to the editor of this journal. Mr. Stead has prepared an exhaustive paper on the subject, to be read at the May meeting of the Iron and Steel Institute. All those interested in metallography will look forward with the greatest interest to its publication. The paper will be reproduced in full in the July issue of *The Metallographist.*

The industrial importance of Mr. Stead's statement will not escape the reader. If indeed he has made it possible to readily locate, under the microscope, the phosphides and sulphides, always present in commercial irons and steels, a new era may truly be said to have begun for the science of metallography, which will probably be characterized by a greatly increased use of the microscope in the steel industry.

The Crystalline Structure of Metals. — The study of the physical constitution of metals and metallic alloys by the employment of the microscope in connection with etching processes continues to add much to the engineer's stock of information concerning the reasons for the behavior of such material under various conditions, and one of the latest contributions to this branch of science is the Bakerian lecture of Professor J. A. Ewing before the Royal Society.*

Most of the researches in metallography which have thus far been made relate to the physical constitution of the metal after

* Professor Ewing's and Mr. Rosenhain's paper is published in full in the present number of *The Metallographist.*

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it has been cast, wrought, or otherwise prepared for use, but this paper discusses the effect produced upon the structure by the application of stresses, and consequently undertakes to reveal the nature of what is called strain.

A knowledge of the manner in which the crystalline particles of metals yield should go far to assist in the study of test specimens taken in actual practice, and there is every probability that this branch of metallography will find useful practical application. — *Engineering Magazine,* December, 1899.

The Measurement of High Temperatures. — Messrs. H. Le Chatelier and O. Boudouard have just published a most valuable book on pyrometry intitled "Mesure des Températures Elevées." It constitutes an exhaustive and critical review of the progress of pyrometry from the very first attempts of physicists to the present day. The various styles of pyrometers are described and the physical phenomena upon which they are based are critically studied, with all the competency and clearness to be expected on the part of so learned and accomplished a scientist as Mr. Le Chatelier.

As being of special interest to the readers of this journal, attention is called to the chapter dealing with the thermo-electric pyrometer to which the name of the author is justly attached. This chapter is subdivided as follows:

Principle. — Experiments of Becquerel, Pouillet and Regnault. — Experiments of H. Le Chatelier. — Heterogeneity of the wires. — Selection of the couples. — Methods of electrical measurements. — Method by opposition. — Galvanometric method. — Resistance of the couples. — Galvanometers. — Various types of galvanometers. — Disposition of the wires of the couples. — Junction of the wires. — Insulation and protection of the wires. — Cold junction. — Graduation. — Formula. — Fixed points. — Experimental results.

The various recording arrangements which have been used in connection with the Le Chatelier pyrometer are also fully described.

The book will be found very valuable by all those using pyrometers or interested in pyrometry. It is published by Carré & Naud, Paris, contains 115 pages and is neatly bound in flexible cover. The price is 5 francs.

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Metallography and the Steel Industry. — Since the first of the year, a number of steel producers and steel users have, to our knowledge, added a microscopical outfit to the equipment of their laboratory, and numerous inquiries are constantly received. With the exception of the crucible steel makers and the railroads, which are still hesitating, there are now few important steel makers or steel consumers which do not subject that metal to a microscopical examination, or which are not contemplating doing so in the near future. There cannot be any better proof of the increasing industrial importance of metallographic methods.

Measurement of Hysteresis of Iron. — Professor André Blondel has a most ingenious instrument for the measurement of hysteresis of iron. It consists of a horse-shoe magnet adapted to be turned about a vertical axis by a handle and gearing. There is also a delicately pivoted axis carrying at its top a support for holding a cylinder of punched rings of the metal to be tested and the index which plays over the scale. On the lower part of the vertical pivot of the apparatus is a helical spring. When the handle is turned and the magnet spun round there is a corresponding tendency for the bundle of rings to turn in the same direction. Readings are taken by swinging the magnet through one complete revolution slowly or rapidly.—*Industries and Iron,* Feb. 24, 1899.

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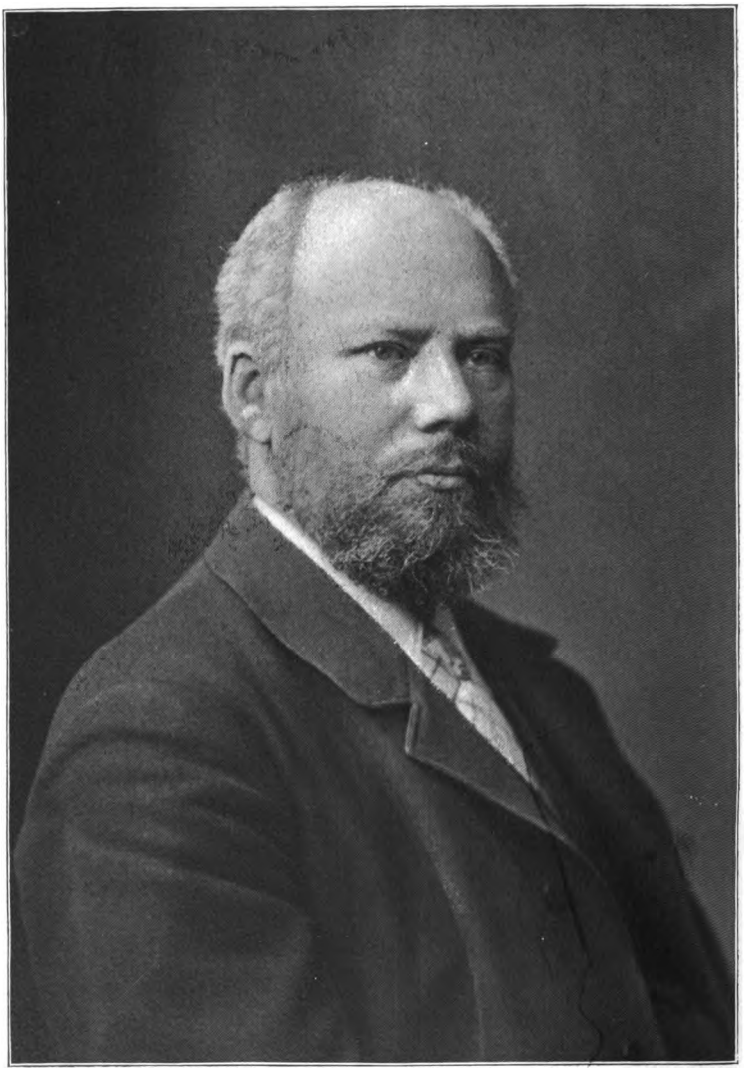

ADOLF MARTENS

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The Metallographist.

A QUARTERLY PUBLICATION DEVOTED TO THE STUDY OF METALS, WITH SPECIAL REFERENCE TO THEIR PHYSICS AND MICROSTRUCTURE, THEIR INDUSTRIAL TREATMENT AND APPLICATIONS.

Vol. III. JULY 1900. No. 3.

ADOLF MARTENS.

ADOLF MARTENS, who was the first after Dr. Sorby to take up the microscopical examination of iron and steel, was born at Backendorf in Mecklenburg-Schwerin, March 6, 1850. He went to the "Realschule" at Schwerin and then worked for two years in a machine shop as a locksmith, cabinet-maker and founder. From 1868 to 1871 he studied at the Industrial Academy of Berlin, now the Technical Institute. He was then engaged for twelve years as a bridge engineer by the Prussian State R. R. His duties in that capacity brought him in close contact with the iron and steel industry of Westphalia and Silesia, as much of the bridge material used by the Prussian State R. R. was purchased there. The process of the manufacture and of the working of steel greatly interested him, and to the study of these subjects he devoted most of his spare time. Prof. Martens says that his interest was greatly stimulated by a small book by E. Schott published in about 1860 on the art of founding, and in which the microscopical examination of iron was advocated.

In 1875 Prof. Martens took up his microscopical work, being then completely ignorant of Dr. Sorby's previous researches in the same field. His first paper on the subject was published in 1878, and was followed by numerous articles dealing with the metallography of iron and steel, a complete list of which is given below:

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On the Microscopical Examination of Iron. — *Zeitschrift des Ver. Deutscher Ing.*, Vol. XXI, pages 11, 205, 481, January, May and November, 1878; Vol. XXIV, page 397, August, 1880. With numerous illustrations. Translated in *Engineering*, Vol. XXVIII, pages 88-90. Thirteen illustrations. Abstd. in *J. Iron and Steel Inst.*, Vol. II, 1879, page 608.

Presence of Defective Structure in Rail Steel. — Glaser's *Annalen für Gewerbe und Bauwesen*, December 15, 1880, pages 476-7.

The Microscopic Structure and Crystallization of Pig Iron. — *Zeitschrift des Ver. Deutscher Ing.*, Vol. XXIV, page 397. Abstd., *J. Iron and Steel Inst.*, Vol. I, 1881, pages 344-5.

Microscope for the Investigation of Metals. — *Stahl und Eisen*, Vol. II, pages 423-5, September, 1882.

On the Microscopical Examination of Iron. Seven illustrations. Both apparatus and structure described. — *Verhandl. des Ver. zur Beförderung des Gewerbfleisses*, 1882, page 233. Translated in *The Iron Age*, Vol. XXX, No. 18, pages 1, 9, November 2, 1882.

Description of a collection of 120 sections, illustrating the microscopical structure of different kinds of iron and steel belonging to the "Bergakademie zu Berlin." — Berlin, 1884.

The Microscopical Structure of Wrought Iron and Steel. — *Stahl und Eisen*, Vol. VII, pages 235-42, April, 1887. — Abstd. in *J. Iron and Steel Inst.*, Vol. I, 1887, page 449.

On the Microscopic Examination of the Structure of Iron. — *Stahl und Eisen*, Vol. IX, pages 393-6, May, 1889.

Microscopic Equipment of the Royal Mechanical Technical Experimental Inst. — *Mittheil. aus den Kgl. Technischen Versuchsanstalten*, 1891, Heft 6, page 278.

The Structure of Rail Heads. — *Stahl und Eisen*, Vol. XII, pages 406-15, May 1, 1892. — Abstd. in *J. Iron and Steel Inst.*, Vol. II, 1892, pages 498-9.

The Microscopic Examination of Iron (Relates to Wire). — *Mitth. aus den Kgl. Techn. Versuch.*, Part II. *Stahl und Eisen*, Vol. XII, page 672, July, 1892. Abstd. in *J. Iron and Steel Inst.*, Vol. II, 1892, pages 485-6.

On several microscopic examinations of iron made at the Mechanical Technical Experimental Institution. Two plates. — *Mitth. aus den Kgl. Techn. Versuch.*, Vol. X, pages 57-68, 1892, Heft 2.

Microscopic Examination of Metals. — Glaser's *Annalen*, Vol. XXX, page 201, 1892.

Microscopic Investigation of Metals. — *Verhandl. des Vereins für Eisenbahnkunde*, 1892, page 67. Abstd. in *J. Iron and Steel Inst.*, Vol. I, 1893, page 365. Describes methods and apparatus used at the Berlin Royal Institute for Microphotography.

The Microstructure of Ingot Iron in Cast Ingots. — Trans. *Amer. Inst. Min. Eng.*, Vol. XXIII, pages 37-63, 1893. Abstd. in *J. Iron and Steel Inst.*, Vol. II, 1893, page 489. Abstd. in *Eng. and Min. J.*, Vol. LVI, pages 217-9, 241-2, 265.

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The Microscopy of Metals at the Engineering Congress at Chicago, 1893. Two tables, 47 illustrations. — *Stahl und Eisen*, Vol. XIV, pages 758-69, September, 1894.

Reproduces 57 illustrations of the microphotographs of various kinds of iron, originally shown at the Chicago Exhibition. — *Mitth. aus den Kgl. Techn. Versuch. zu Berlin*, Vol. XI, page 247. Abstd. in *J. Iron and Steel Inst.*, Vol. II, 1894, page 442.

Discussion between A. Martens and A. Sauveur on "Ferrite" and "Pearlite." — *Stahl und Eisen*, Vol. XV, pages 537-9, June, 1895. Abstd. in *J. Iron and Steel Inst.*, Vol. II, 1895, pages 544-5.

Criticism of Osmond's work. — *Stahl und Eisen*, Vol. XV, pages 954-7, October, 1895. Abstd. in *J. Iron and Steel Inst.*, Vol. I, 1896, pages 487-90.

Report of the Subcommission No. 15 of the fourth permanent Commission for an agreement on uniform methods of testing building and construction materials, to determine the importance of the method of examining the microstructure of metals. Investigation of the possibility of a uniform method of examination. Proposition. Zurich, 1895.

Researches upon the influence of the degree of heat in rolling, upon the properties of strength and the microscopic structure of ingot iron rails. — *Mitth. aus den Kgl. Techn. Versuch.*, Vol. XIV, Heft 2, page 89, January, 1896.

When Prof. Martens first undertook his metallographic work, he could only devote to it his spare time, and he had to pay himself for the needed apparatus.

In 1884 Prof. Martens was appointed director of the Royal Mechanical Technical Experimental Station, then newly founded at Charlottenburg, Germany, — a position which he still occupies.

Upon entering into his new position, it was his hope to be able to carry on his metallographic work more thoroughly and systematically, but that department of researches was assigned to the sister Institute, and Prof. Martens had to continue his investigations at his own expense, and could only devote to them such time as he could spare. His other duties, moreover, soon became so exacting that he had to abandon altogether his metallographic inquiries, although the work had now been assigned to the Institute of which he was the head, entrusting Mr. E. Heyn, a member of his staff, with their continuance. Our readers are familiar with some of the good work already published by Mr. Heyn.

Although Prof. Martens' written contributions are sometimes laborious, they always testify of painstaking, methodical

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work thoroughly done. He has repeatedly contended that metallographists should devote their energy to a thorough investigation of the best methods of developing the structure of the polished metallic surfaces, and that they should for a while content themselves with a description of what they see, avoiding hasty inferences.

In his writings Prof. Martens has adhered to these precepts, for his articles have been mostly of a descriptive character, often perhaps too minutely so, and he seldom draws any conclusions, or advances any theory.

If we may be permitted some criticism, we would venture to say that the fear of hasty theories, while generally a very wholesome one, may yet be carried too far, and such seems to us to have been the case with Prof. Martens. When experimental facts have been recorded, sufficiently numerous and concordant to warrant the putting forward of a theory, it is right and desirable to do so, for whether eventually proven false or true, it will suggest inquiries which cannot but help in advancing our knowledge.

If, on the contrary, the experimental evidences be left scattered and no effort made to connect them under some theory, they will be productive of little good.

Prof. Martens carried to a high degree of perfection the technology of the subject, as evidenced by the excellent illustrations which accompany his papers, and his equipment at Charlottenburg is probably the most elaborate and complete in existence.

We can only briefly allude here to Prof. Martens' activity and accomplishments in other lines of researches. Most of our readers are undoubtedly acquainted with the international reputation of the Experimental Institution of which Prof. Martens is the head. Started in 1884 with a personnel of four, it has now a staff of 88 members. It is exclusively an experimental and testing station divided into four departments dealing respectively with the testing of metals, of building materials other than metals, of paper and of oil. Numerous valuable papers on these subjects have been published in the "Mittheilungen" of the Institute, and reproduced and criticised in many technical journals.

Such gratifying development of this Institution and the very

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creditable work carried on, is undoubtedly greatly due to the incessant activity and to the enthusiasm of Prof. Martens.

Prof. Martens devoted much time to the development and perfection of testing machinery and mechanism, and his written contributions to the various subjects which he investigated number nearly ninety.

Much of his original work and of the mechanical improvements which he introduced in the art of testing will be found in his recent work, "Hand-Book of Testing Materials," very recently translated into English by G. C. Henning, and which, in spite of too great adherence to the German text on the part of the translator, and of the deplorable mistake of placing all the illustrations by themselves in a separate volume, will undoubtedly be received with much appreciation by English-speaking engineers and metallurgists.

Prof. Martens is vice-president of the International Association for Testing Materials, and has taken a very prominent part in all the meetings held by the association. He is chairman of the German association for the testing of materials and a member of numerous technical societies.

Although, as already stated, Prof. Martens is no longer personally engaged in metallographic researches, he continues to follow their progress with the greatest interest, and his experience and faith in the importance of the subject will undoubtedly be an inspiration, and greatly contribute to the success of the metallographic work carried on under his management and guidance.

ON THE CRYSTALLOGRAPHY OF IRON.*

By F. OSMOND.

Definitions.

AT the outset of a study on crystallography, it is not superfluous to ask: "What is a crystal?" The answer is not as easy as might at first be deemed.

Haüy† says in substance, that the name of crystals is given

* *Annales des Mines*, January, 1900.

† *Traité Elémentaire de Physique*, Vol. I, page 60. Paris, 1803.

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to those bodies which result from the attraction of molecules, as the latter are given up by a liquid or otherwise.

According to Beudant, "crystals are geometric polyhedra."*

The definition of Mallard remains nearly identical to that of Haüy: "When a substance passes, sufficiently slowly, to the solid state, it generally assumes a regular geometric form and is then called a crystal."†

Friedel wrote more recently still: "Instead of existing in irregular fragments, a mineral may possess regular geometric forms which observation proves to be perfectly constant, and which, owing to this constancy, are well adapted to serve to define the minerals."‡

De Lapparent§ explains rather than defines: "The species in mineralogy can only be defined through its molecule."

"The definition of the *species* being based upon the composition and the structure of the molecule or *atomic fabric,* the *variety* must be defined by the structure of the whole, that is, by the *molecular fabric.*"

"... It is for the mineralogist to decide about the *form* of atomic structures, or, at least, about their degree of symmetry, because this symmetry is found most distinctly in the varieties called *crystallized,* and whose most marked character consists in external forms susceptible of a geometrical definition.

"The *crystalline* state is that in which the arrangement of the molecules is *regular* ... Crystallized substances assume externally plane faces with geometrical outlines ..."

Summing up, it is seen that the French writers on crystallography have been in accordance for a century, but it may be said that these definitions, isolated from the commentaries accompanying them, do not all fulfill the two required conditions of including all that is defined and only that which is defined.

When it is said, for instance, that crystals are geometrical polyhedra, it must be understood or further explained, as is, indeed, generally done in most classical treatises, that these polyhedra have been produced spontaneously, differing in this from

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* *Minéralogie,* page 11. Paris, 1851.

† *Cristallographie* (*Encyclopédie chimique,* F. Frémy, Vol. I, page 609). Paris, 1882.

‡ *Cours de minéralogie,* page 2. Paris, 1893.

§ *Cours de minéralogie,* page 7. Paris, 1884.

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the polyhedra artificially cut, and that their external forms must be a function of their internal symmetry, so as to distinguish them from pseudo-crystals.

It does not seem difficult, however, to express simply and synoptically the above definitions.

It is sufficient to consider that all solid bodies possess: (*a*) a substance, and (*b*) a form.

(*a*) The substance may be crystallized or amorphous.

If the substance be characterized by the fact that "the properties of the matter possessing an orientation, vary around each point in said direction, and that this variation follows a certain law, which is the same for all the points of the same homogeneous mass," the substance is said to be crystallized. On the contrary, if "the properties are identical in all directions around each point," the substance is said to be amorphous.*

(*b*) The form may or may not be that of a geometrical polyhedron; in the first case the forms of the polyhedron may or may not be a function of the internal symmetry of the substance.

Six mathematical combinations are, therefore, possible:

1. The substance is crystallized; the forms are geometrical and a function of the internal symmetry of the substance: the substance is a *crystal.*

2. The substance is crystallized; the forms are geometrical, but bearing no relation to the internal symmetry of the substance: the substance is a *pseudo-crystalline solid whose matter is crystallized,* a solid which, according to its external forms, will be called grain, cellule, column, etc.

3. The substance is crystallized; the forms are not geometrical: the substance is a *fragment of crystallized matter* which will be called, according to circumstances, grain, piece, block, etc.

4 and 5. The substance is amorphous; the forms are geometrical: the distinction based upon the fact that the external forms are or are not a function of the internal symmetry is not, evidently, applicable here: the substance is a *pseudo-crystalline solid whose matter is amorphous,* and which will be called, according to circumstances, grain, cellule, column, etc.

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* The lines between quotation marks are from Mr. H. Le Chatelier, *Recherches sur la dissolution* (*Annales des Mines,* February, 1897).

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6. The substance is amorphous; the forms are not geometrical: the substance is a *fragment of amorphous matter,* which will be called, according to circumstances, grain, piece, block, etc.

It has seemed to me desirable to thus formulate and condense these definitions, which, I think, are simple expressions of classical opinions, because they are often lost sight of. In the study of opaque bodies, for instance, chemists frequently affirm that the substance is crystallized because of the nearly plane small faces exhibited by a fracture, while these faces may as well be shrinkage surfaces, as in the case of clay, or may be due to limitations of independent development, such as would be exhibited by plastic grains compressed in a mould, or by small rubber balloons filled in a closed space. Again, owing to the increasing importance rightly attached to optical properties and to the theory of reticular groupings, the external forms have lost their former supremacy, and several authors have come to use the word *crystal* as a synonym of *crystallized matter.*

This leads to confusion, as I had frequently occasion to ascertain while studying the subject of this paper. The pseudo-crystalline grains which make up the mass of soft steel were first called crystals, while the crystallized or amorphous condition of their substance was still unknown, such conclusion being based upon their polyhedrical shape; which was, of course, erroneous. The error once recognized, however, and sometimes to conceal it an effort was made to retain the word *crystal,* under the pretext that, as our knowledge increased, the crystallized condition of the internal substance had been subsequently demonstrated.

Much disorder resulted from this, and Mr. Stead made an effort recently to bring back some clearness and logic to the crystallographic vocabulary of metallurgists.*

In the following pages I shall, of course, conform my expression to the definitions just formulated:

Position of the Question.

The views generally accepted regarding the crystallography of iron are condensed in one line in *La Mineralogie* of Mr. de

* *Journal Iron and Steel Institute,* 1898, II; *The Metallographist,* October, 1898.

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Lapparent (first edition, p. 455): cubic system; forms p, a', $a\frac{1}{2}$, rare cleavages along p or a'.

The question, however, is more complex.

It is known that pure iron may undergo between the ordinary temperature and its melting-point, at least two allotropic transformations: one of them (A_2), progressive and corresponding to the disappearance (or appearance) of ferro-magnetism, takes place, most of it at least, between 700 and 760° C.; the other (A_3) is sudden and takes place at about 860°. Dr. Ball, moreover, has detected a maximum in the hardness of very mild steel corresponding to a quenching temperature of about 1300°,* and Mr. Currie† finds that the magnetic properties of iron, which remains diamagnetic after the disappearance of its ferro-magnetism, undergo a sudden change at that temperature (1300°). These facts might indicate the existence of a third allotropic transformation, and if its existence were proved, a revision of our knowledge would have to follow. As these experimental facts, however, may be interpreted in various ways, and as I have been unable to detect a thermal critical point in the vicinity of 1300°,‡ I shall continue to say, as it is the custom, that iron below A_2 is in the *Alpha*-state, between A_2 and A_3 in the *Beta*-state, and above A_3 to the melting-point in the *Gamma*-state.

The molecular transformations of iron regulate necessarily the crystallography of that metal. It is true that two allotropic forms of the same substance may be isomorphous, as well as two different substances, so that a molecular change does not necessarily imply a change in the crystalline forms, but if iron does crystallize in several systems or in several varieties of the same system, it is certain that these crystalline modifications will correspond to the allotropic modifications.

The problem of the crystallography of iron may, therefore, be formulated as follows:

What are the crystalline forms of iron corresponding to its three states, *Alpha, Beta* and *Gamma?*

As a matter of fact the problem may be a very difficult one to resolve. In the first place, industrial products such as those

* *Journal Iron and Steel Institute,* 1898, II.

† *Thèse,* Gauthier-Villars et fils. Paris, 1895.

‡ *Mémorial de l'Artillerie de la Marine,* second series, Vol. XIX, page 24.

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in which we are most interested are always more or less impure, and the impurities may exert a certain influence; then, at the ordinary temperature, which is the temperature at which our observations are usually made, iron, except in some special cases, has normally assumed again the *Alpha*-state, after having passed, at least once, since its solidification, through the two states stable at high temperatures. When we examine the metal in the cold, therefore, and detect certain crystalline forms, we must ask whether these forms belong to the actual molecular condition or to one of the past molecular states. In short, every experimental fact must be subjected to a critical discussion which is always delicate and which has never been conducted methodically.

Crystallography of Gamma-Iron.

The facts which may serve in ascertaining the crystalline forms of *Gamma*-iron may be classified in five groups:

A. The existence of crystals formed during the solidification of the mass in the cavities of ingots or castings of steel and cast iron.

B. The structure of quenched steel.

C. The structure of manganese steel.

D. The structure of nickel steel.

E. The structure of iron and its alloys in the range of stability of *Gamma*-iron, as revealed by etchings conducted at the proper temperature.

A. Crystallization during Solidification. — The *Gamma*-state, if it be supposed that Ball's point does not correspond to an allotropic modification, is, by definition, the state which iron assumes in solidifying. Our first thought, therefore, must be to turn to the results of the crystallization during solidification in order to study the crystalline forms of *Gamma*-iron, for while subsequent allotropic modifications may change the internal structure, they will leave the external forms of the crystals unaltered.

Pure iron, however, in a cast condition, is not an industrial product, and we must consider it as the limit of industrial products, when foreign matters, especially carbon, are made to decrease progressively.

If we examine the accompanying diagram, in which Sir

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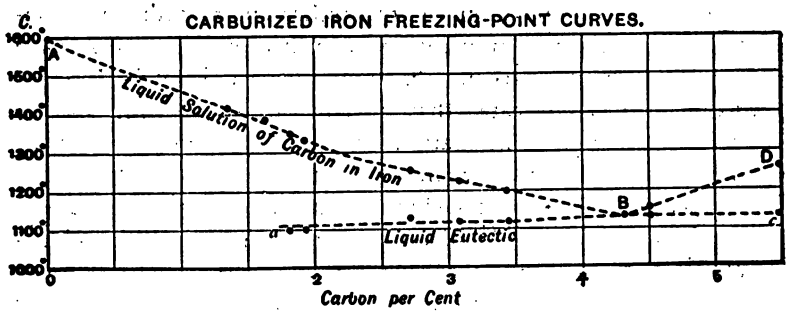
W. Roberts-Austen* has plotted as ordinates the critical temperature of carburized irons, and the corresponding carbon contents as abscisses, we note that the solidification of these alloys takes place at a constant temperature if the percentage of carbon remains below a certain value (a), and at two different temperatures if that carbon content is exceeded. In the first case, the solidified substance is a solid solution of carbon in *Gamma*-iron, which is not saturated at the temperature of solidification; in the second case, the first portion to solidify is still that solid solution, but which is now saturated at the temperature of solidification, and the second portion to solidify is a eutectic alloy of that same solution and of graphite, if the cooling be very slow;



of that solution and of cementite (Fe_3C) if the cooling be more rapid, without being very rapid.

From the above considerations, carburized irons are naturally divided into two classes: those of the first will have only one solidification point; those of the second class will have two points of solidification. This classification corresponds to the ordinary distinction between cast iron and steel; at the other end is found iron proper which may form a third class.

a. *Cast Iron.* — Sir Roberts-Austen's samples contain only traces of silicon and phosphorus, and it may be asked what would be the curves of solidification in the case of commercial products which always contain silicon and frequently phosphorus in relatively large proportion? This question is all the more important

* Fifth Report to the Alloys Research Committee; Institution of Mechanical Engineers, February, 1899. — Also *The Metallographist*, Vol. II (1899), page 186.

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that these two impurities cause the disappearance of the point A_3, maintaining the metal in the *Beta*-state up to the melting-point. Unfortunately, this complete study of the question is still to be made; the examination of a few samples,* however, has shown that carbon causes the reappearance of the point A_3 in cast iron containing silicon and phosphorus, and that grey cast iron usually exhibits at least two solidification points, if they are pure, and three at least if they are phosphoritic. In every case the constituent solidifying first is always *Gamma*-iron, saturated in the solid state with carbon, silicon and eventually phosphorus; most of the silicon appears to be concentrated in the constituent of second solidification, and phosphorus in that of third solidification.

We may, therefore, in our researches on crystallization, consider grey commercial iron jointly with theoretical cast iron made up only of iron and carbon. The mere inspection of Sir Roberts-Austen's curves indicates that crystals of *Gamma*-iron may be found in cast iron, in the natural cavities (those produced by the contraction of the mass during solidification: pipes), or in artificial cavities (those produced by the partial pouring of a solidifying mass), or again embedded in products of subsequent solidification, where they will be detected by metallographic methods.

The presence of crystals in the pipes of castings has long been known and has been described on many occasions.

Modern books on the synthesis of minerals, such as that by Messrs. Fouqué and Michel Levy,† as well as Mr. Bourgeois'‡ work, give many instances borrowed from Fuchs.§ But in Fuchs' bibliography the French mineralogists of the 18th century have been ignored.

Among them, however, there is one at least, Mr. Grignon, ironmaster and correspondent of the royal Academy of Sciences, whose remarkable observations should not be forgotten, and

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* *Annales des Mines*, eighth series, page 39.

† *Synthèse des Minéraux et des Roches*, page 365. Paris, G. Masson, 1882.

‡ *Reproduction Artificielle des Minéraux*, page 21. Paris, Dunod, 1884.

§ *Die künstlich dargestellten Mineralien.* Extract from *Natuurkundige Verhandelingen van de Hollansche Maatschappij der Wetenschappen te Haarlem*, 1872.

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whose curious drawings I am pleased to be able to reproduce here, owing to the kindness of Mr. Fremont, who placed at my disposal one of the rare books of his fine technological library.*

"Grey cast iron," says Grignon in his paper entitled "Réflexions chymiques et physiques sur les métamorphoses du fer" (page 71), "when in a perfect condition gives a very regular crystallization, every crystal being distinct and isolated; but to obtain it, the metal must be cooled very slowly during several days, the contraction must be considerable, and the phenomenon must take place without disturbance; each crystal is then a kind of pyramid whose base is a rhomb, and along each side of which are attached at right angle other pyramids whose bases are equal to the diameters of the point of incidence of the principal pyramid to which they are attached; and as the diameters decrease gradually, the lower pyramids are bigger and longer, the upper ones shorter and more slender, there being a true proportion between the diameter of the base and the length of the column. The four cruciately opposed pyramids are identical, and are attached by their bases, and in symmetrical gradation of distances, lengths and sizes, to a line supposed straight and oblique to the top of the central pyramid, from whose very sharp apex may be seen all the apices of the lower pyramids, and which constitutes a crystal of cast iron regularly grouped. The small cavities in which a large number of these small crystals are found, present, under a magnifying glass, the appearance of a small metallic forest, made up of trees with quaternary branches. Each pyramid is composed of a series of rhombs whose sides are inclined, thus giving a larger surface, which is attached to the smallest side of the rhomb which supports it, etc." (See Figs. 1 to 4.)

In another paper† whose title alone indicates that the author foresaw the work of the synthetic mineralogists, Grignon describes some crystallizations of cast iron and of iron more perfect than the first ones. "It is," says he, "a fragment which was embedded in a mass of cast iron and of slag, kept melted for several days, and whose cooling lasted several days in a furnace, so that the surrounding substance had time to shrink and the molecules of cast iron time to assume their regular crystalline

* *Mémoires de physique.* Paris, Delalain, 1775.

† *"Sur des Crystallisations métalliques, pyriteuses et vitreuses artificielles formées par le moyen du feu."*

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form. The piece is irregular as a whole ... The middle portion rises like a crest; it is made up of numerous crystals of cast iron and of iron. Each crystal is composed of several others, regularly grouped. The first element is a rhomb upon which rest perpendicularly other rhombs, decreasing in size until they form a pyramid with a rhomboidal base. Upon the four sides of this central and main pyramid, other pyramids of similar form are attached at right angle, which decrease in length and size according to the position they occupy; so that the section of such a compound crystal is a quandrangular star, while the profile of its elevation seems to be composed of small fir trees, with quaternary branches. Some of these crystals have an infinite number of parts, that is, they are groups of complete and regular crystals attached to the lateral pyramids of the first crystal. These crystals are all absolutely identical; they are regular in all their parts; if some of them differ from each other, it is only with regard to their size; they are, therefore, perfect." (Figs. 5 to 7.)

In brass also are found similar crystals. (Figs. 8 to 11.)

Buffon also was an ironmaster, and a complete set of his writings include some papers on metallurgy which are not the least interesting portion. He devised, in collaboration with Guyton de Morveau, an experiment to cause cast iron to crystallize; "the trial," he says,* "was conducted in a very large crucible of molybdenum† with a mass of cast iron of about 250 pounds: a hole eight or nine inches in diameter had been provided near the bottom of the crucible and had been plugged with earth (*terre de coupelle*); the crucible was placed on a grate and its lower part surrounded by burning coals, while its upper part was protected from the heat by a circular table made of bricks; the crucible was then filled with molten cast iron, and when the surface of the bath which was exposed to the air began to solidify, the hole at the bottom of the crucible was quickly opened, and more than half of the metal being still liquid, escaped, leaving a large cavity in the interior of the mass; this cavity was found to be covered with some very small crystals, showing under the magnifying glass some octahedral faces; most of them, however,

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* *Œuvres complètes.* Verdière et Lagrange, 1826, Vol. VIII, page 247.

† Molybdenum was then a synonym of plumbago.

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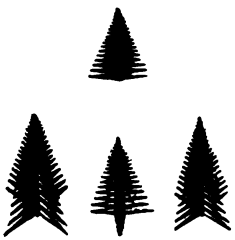

Fig. 1

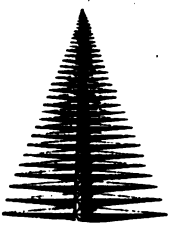

Fig. 2

Fig. 3

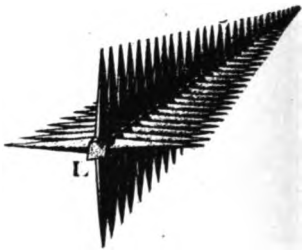

Fig. 4

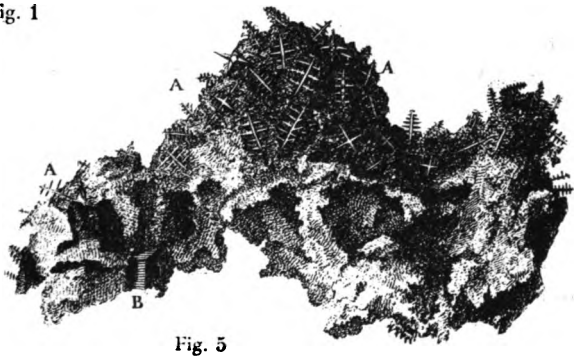

Fig. 5

Fig. 6

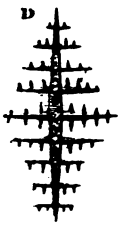

Fig. 7

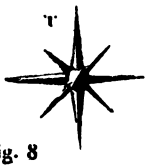

Fig. 8

Fig. 9

Fig. 10

Fig. 11

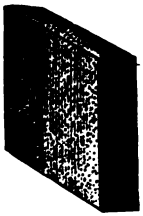

Fig. 29

Fig. 26

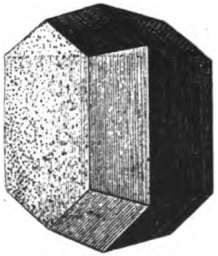

Fig. 27

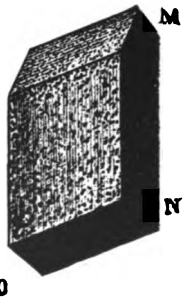

Fig. 30

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could be detached with a quill, separating into small leaves like mines of iron rich in mica; this, however, is far from the beautiful crystallizations of Grignon, and indicates that in this experiment the cooling was still too rapid."

Bergman also was aware of the octahedral crystallization of cast iron.*

These observations made so long ago, and confirmed many times, have been completed in the last twenty years by chemical analysis and by the examination of polished surfaces.

It will suffice to give a few instances.

Mr. A. Boardsley† found some octahedra nearly one-half inch in diameter in the cavity of some grey cast iron from Gartsherrie. An analysis made by Collins gave the following results:

Iron 93.50 per cent; Carbon 3.11 per cent; Silicon 1.35 per cent; Titanium 0.76 per cent; Phosphorus 0.15 per cent; Manganese, traces.

The carbon was in the graphitic state in the interior as well as at the surface of the crystals.

Prof. Martens described and sketched on several occasions, in elevation and in section, crystals found in the cavities of grey cast iron. They are, he says in substance,‡ skeletons of octahedra. They are composed of parallel teeth, attached normally to the principal axis in two planes at right angle to each other, (Fig. 12). This formation continues frequently to the third and fourth degree. The further it is carried on, i.e. the more numerous the needles of the second, third, etc., order, the more the crystalline skeleton approaches the form of a true octahedron. The isolated teeth are not always needle-like at their extremity, for they may be rounded, (Fig. 13).

Prof. Ledebur has kindly given me a portion of a beautiful specimen found in the cavity of the sinking head of an anvil of phosphoritic cast iron. Fig. 14, drawn by Mr. Fremont, shows its appearance when magnified about 4 diameters: numerous crystallites may be detected, most of them being skeletons of octa-

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* *Opusculcs chymiques et physiques,* translated by de Morveau. Dijon, 1780, Vol. II, page 17.

† *Min. Mag.,* No. 11, page 223, and No. 12, page 261.

‡ *Zeitschrift des Vereines deutscher Ingenieure,* Vol. XXII, page 11, and Vol. XXIX, page 397.

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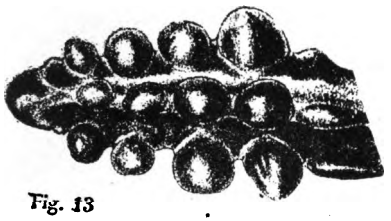
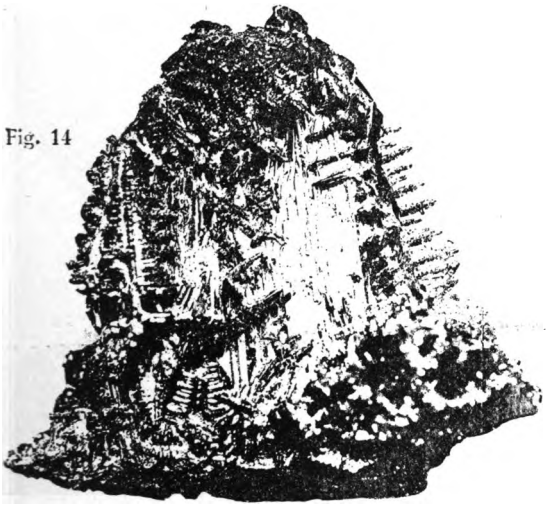


Fig. 13

Fig. 12

Fig. 14



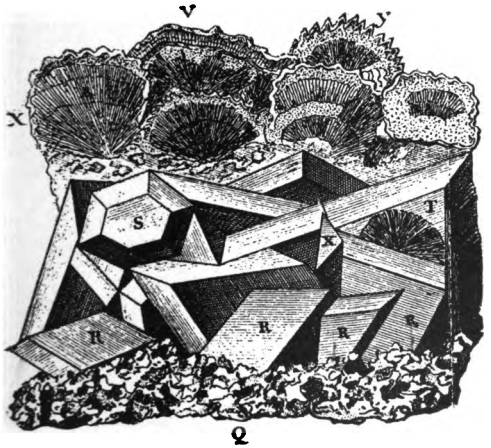


Fig. 28

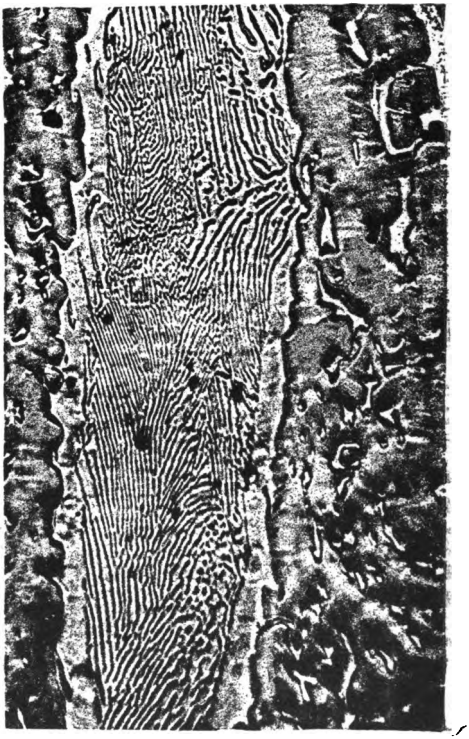


Fig. 15

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hedra; others, especially the main one at the right of the figure, have no visible pyramidal extremities. Fig. 15 shows the appearance of a section of a branch of crystallite, after polishing in relief upon a piece of wet parchment slightly covered with rouge: from the solid solution of carbon in iron, of which the substance of the crystallite was originally composed, the two constituents, iron and the carbide Fe_3C, separated during the point Ar_{321}, forming what has been called pearlyte. Around this portion we see some free cementite and a phosphide which was not resolved by the polishing operation.

White cast iron yields crystals similar to those of grey iron, provided, of course, it does not contain more carbon than the eutectic (about 4.30 per cent), and therefore possesses two points of solidification.

A sample of refined cast iron, for instance, described by Dr. Sorby,* exhibited some rectangular crystallites of pearlyte.

I heated two bars of steel containing 1.70 per cent of carbon in the tube of a Leclercq furnace, with charcoal, in such a way that one of the extremities of the bars placed in the center of the furnace was heated to 1300° C., while the temperature decreased gradually towards the other extremity. In these conditions liquid cast iron was produced at the hottest portion, and in the portion which did not melt I had, in a very short space, the whole series of carburized irons, from the saturation point at the temperature of the experiment, down to 1.70 per cent of carbon, which was the original carbon content of the bars. One of the bars was quenched in ice water, and the other cooled in the furnace.

Fig. 16 shows the appearance of a polished section of the hot end of the bar slowly cooled: it was etched for 10 seconds with nitric acid diluted four times with water; it is magnified 20 diameters. Three regions are easily distinguishable: the extreme end uniformly dark is grey cast iron with graphite minutely divided, and whose structure cannot well be made out, because the mode of preparation of the section was not well adapted to its resolution and the magnification is too low. Then comes a region made up of rectangular and triangular dark crystallites on a light background. It represents white cast iron whose carbon

* *Journal Iron and Steel Institute*, 1887, I, page 278, Fig. 16.

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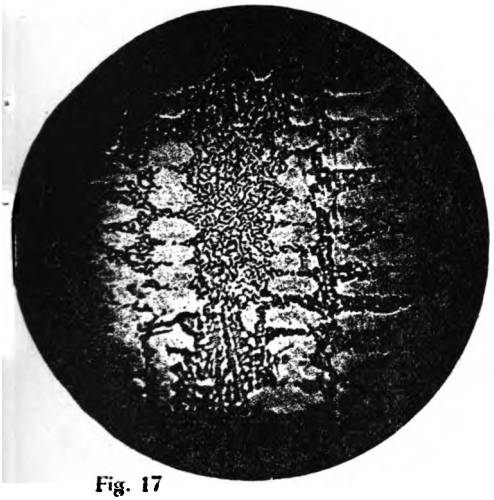

Fig. 17

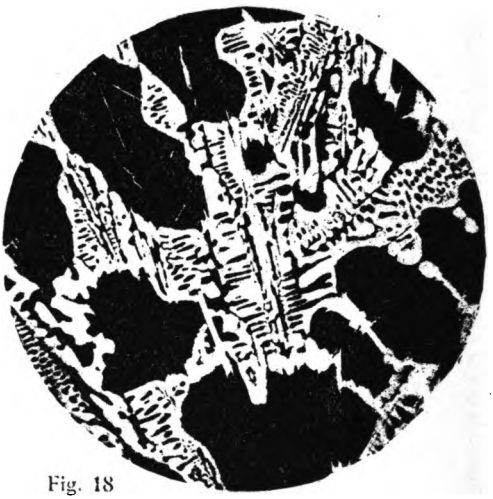

Fig. 18

Fig. 16

Fig. 21

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content decreases from the outside to the center; the crystallites represent the solid solution of first solidification, from which, during cooling, plates of cementite separated first, then the eutectic, pearlyte or sorbite (the latter if the melting of the cementite was sufficiently complete); the light background represents the product of second solidification, that is, another eutectic, that of the liquid solution, whose two components are cementite and that same solid solution composing the first portion. Finally, we gradually return to the primitive steel, which was not altered, and which is made up of grains of pearlyte surrounded and cut by plates of cementite.

Fig. 17 shows a portion of the same preparation magnified 100 diameters, after polishing in relief. The rectangular crystallites of first solidification are depressed and appear light in the photograph; the background is the eutectic made up of cementite and the solid solution, and in which are found some dark dots representing minutely divided graphite ("invisible graphite" of Forquignon, "graphitische Temperkohle" of Ledebur), as the spot represented was situated on the borders of the grey cast iron end of the bar.

The details of this eutectic may be seen in Fig. 18, magnified 250 diameters. The preparation was etched during 10 seconds with nitric acid diluted with four times its volume of water. The light portions are cementite, the dark ones pearlyte or sorbite.

Figs. 19 and 20 represent similar regions of the quenched bar.

The general dispositions remain the same, only the solid solution of first solidification instead of breaking up into cementite and a eutectic mixture of cementite and of ferrite, yields a mixture of two constituents, which have not yet been mentioned here, one of them hard like ordinary quenched tool steel (martensite), the other softer and resembling maganese steel (austenite).

Fig. 19 shows a very good instance of octahedral crystallites. The section was etched with tincture of iodine and magnified 100 diameters. Fig. 20 shows the details under a magnification of 250 diameters.

b. *Steel.* — From the above facts, can it be inferred that the octahedron is the crystalline form of *Gamma*-iron? Not yet, since the octahedral skeletons detected were not at the instant of their solidification made up of pure *Gamma*-iron, but of *Gamma*-

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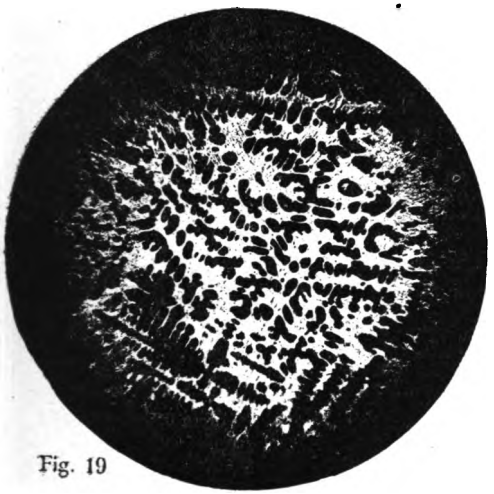

Fig. 19

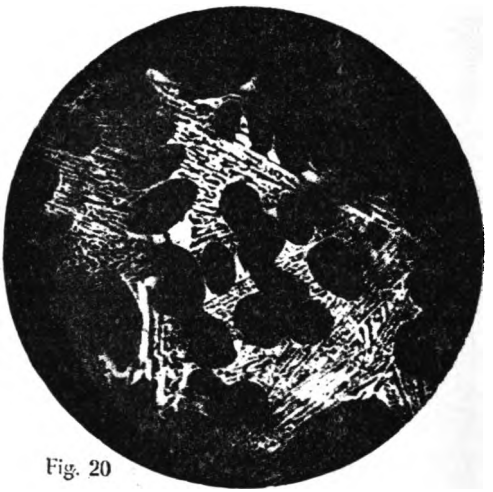

Fig. 20

Fig. 50

Fig. 23

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iron saturated, in the solid state, with dissolved carbon, to say nothing of silicon and phosphorus.

Steel always contains very little phosphorus, seldom much silicon, and in the case of Bessemer and Open Hearth steel, which is produced in such enormous quantity, it contains an amount of carbon much too small to saturate the iron at the temperature of solidification. It does not have, like cast iron, several points of solidification, but its solidification, especially in the case of large ingots, requires time, and crystals may be formed in the cavity existing in the portion which solidifies last.

As a matter of fact such crystals have been discovered many times, notably by Messrs. Tschernoff,* Gautier,† Martens‡ and Fulton.§

Mr. Gautier's crystals were found in the sinking head of a sand casting of steel containing not over 1.30 per cent of impurities in all. They did not present any plane sides, but only octahedral edges much more acute at first sight than a true octahedron. In spite of these deformations, however, they may probably be attributed to the cubic system.

Mr. Valton had the kindness to give me a beautiful sample of similar origin as that of Mr. Gautier's. Fig. 21, on a preceding page, shows one of the principal crystals magnified 6 or 7 diameters; the structure is identical to that of the crystals of cast iron described in the foregoing pages.

Prof. Howe gives in his *Metallurgy of Steel* the reproduction of a remarkable specimen, given to him by Mr. Fulton.

The finest known collection, however, is probably that of Prof. Tschernoff. Fig. 22 shows the appearance of a beautiful specimen, about half natural size. It resembles a crystal of alum.

In short, all steel crystals, whatever their dimensions and their perfection, are made up, like those of cast iron, by successive ramifications parallel to three rectangular axes; their limits are heaps of interlocked octahedra, whose principal axis coincides with the three axes of the system. The extremities are sharper than

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* *Proceedings Institution Mechanical Engineers,* London, January 1880, page 152.

† *Bulletin de la Société Minéralogique.* Vol. II, page 210, 1879.

‡ *Stahl und Eisen,* February 1887.

§ *Metallurgy of Steel,* page 148, Fig. 25.

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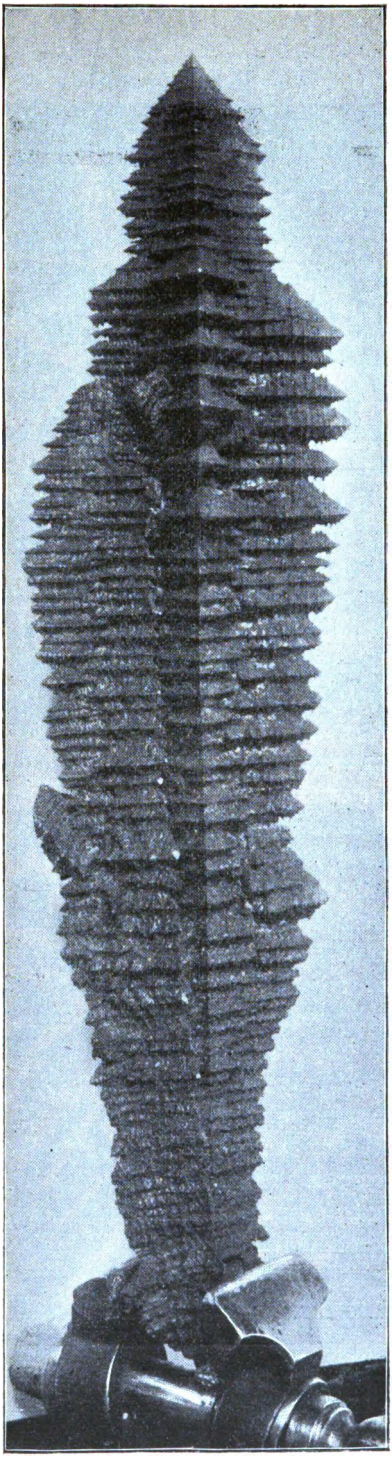

Fig. 22

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those of a true octahedron, but as these extremities represent the envelope of a row of interlocked crystals, there is no reason why each individual crystal should not be a perfect octahedron, although it is difficult to ascertain it by direct measurements.

The sections moreover belong to the cubic system.

Prof. Martens* has reproduced under a magnification of 10 diameters a section through a steel ingot of medium hardness; crystallites may be found whose branches are normal to the principal axis.

The same investigator has published an important paper,† in which many crystallites of the same type are shown, and which are especially distinct after hardening and tempering. The carbon content varied between 0.32 and 0.50 per cent.

Prof. Tschernoff had the kindness to give me a fragment of a large crystal such as is represented in Fig. 22. I prepared a section of it perpendicularly to the principal axis; the ferrite is oriented, except for secondary interference, in directions parallel to the two transversal axes, dividing the pearlyte into areas roughly rectangular (Fig. 23, on a preceding page); the preparation was etched with concentrated nitric acid, and magnified from 6 to 7 diameters.

Sections perpendicular to the transversal axis have the same appearance.

Figs. 24 and 25 show the structure of the sound region of the sinking head of a steel casting containing about 0.45 per cent of carbon. The white lines (ferrite) cut each other at right angles or more frequently form equilateral triangles, which suggest the figures of Widmanstaetten, in which crystallographers detect the characteristics of the regular octahedron.

We are led therefore to infer that *Gamma*-iron possesses both cubic and octahedral cleavages. In the sections of medium hard steel which we have just examined (Fig. 23, 24 and 25), the separation into ferrite and pearlyte does not exist at the instant of solidification; the carbon was then disseminated through the whole mass. It might be argued that ferrite assumed its individuality in passing to the *Beta*-state, that the carbon was driven off by the crystals of *Beta*-iron, and therefore, that the

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* *Mittheilungen aus den Kön. tech. Versuchsanstalten,* 1891.

† *Transactions Am. Institute Min. Eng.,* Vol XXIII, page 37.

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Fig. 24

Fig. 25

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actual internal arrangement does not correspond to the initial arrangement in the *Gamma*-state. Since, however, in Prof. Tschernoff's crystal the internal symmetry of the network of ferrite is evidently related to the external forms, which are undoubtedly those of *Gamma*-iron, it must be that that internal symmetry is also that of *Gamma*-iron, or that *Gamma*-iron and *Beta*-iron both crystallize around the same axis, an hypothesis which will be discussed further on.

c. *Iron.* — It remains to be seen, whether the similar crystalline forms which *Gamma*-iron assumes in cast iron and steel, still persist in the absence of carbon.

Lacking carbonless iron in a cast condition, we may turn to burnt iron for information. Unfortunately most of the observations which have been recorded relate to samples whose composition is not given or only imperfectly.

Grignon is here again the first author who appears to have defined burnt iron.*

"When the end of a *marquette d'encrenée*,† which is coarse (*crude*)‡ iron, is exposed to a temperature sufficiently high to melt the slag which it contains internally, allowing the molecules of iron with which it was united to come in contact, the iron assumes, in that slag which was its dissolvant, and which acts as the vehicle of its crystallization, geometrical forms. If the temperature be very high the bar of iron cracks under the hammer and agglomerations of crystal are detached, called *grumillons* by blacksmiths. (See Fig. 26 on a preceding page.)

"I found in chunks of slag expelled by the hammer large agglomerations of such crystals which were expelled with it, and which had crystallized in the slag as in their natural solvent. Slag is to crystals of iron what water is to saline crystals . . .

"These crystals of iron are seldom very regular, because the heat to which they owe their existence welds them together, and alters their angles through the action which it has upon their sub-

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* *Opere citato,* page 79.

† A "*encrenée*" is the first shape given to a bar of iron. The mass is forged down in the middle to the dimensions of the finished bar, leaving both ends unforged. A "*marquette d'encrenée*" is an *encrenée* with one of the ends forged. These definitions are given by Grignon.

‡ That is imperfectly freed from its slag and possibly still somewhat carburized.

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stance. The most perfect among them appeared to be polygonal hexahedra made up of several rhomboids united by their large sides." (See Fig. 27 on a preceding page.)

This old description is interesting for several reasons. Although the opinion that slag was a solvent of iron, has been shown to be inaccurate, it contains nevertheless, in germ, one of the most fruitful methods of mineralogical synthesis followed in our century. Another of his views which consists in considering the grains of burnt iron like true crystals, has been held to the present time, and is probably still accepted by some. Fig. 27 drawn by Grignon, must have been, if we consider his description so full of reserves, idealized, or rather geometrized; it must represent a conception rather than a candid reproduction. As a matter of fact, the grains of burnt iron are pseudo-crystals, and their forms, which are roughly geometrical, are the result of the mutual limitation of contiguous crystalline developments proceeding from independent centers. Prof. Wedding* justly remarks, that their faces are generally pentagonal; they are indeed pentagonal dodecahedra, such as are produced, more or less altered, by the compression in a close vessel of plastic spheroids piled up at random. It is, however, not at all unlikely that at the surface and in favorable conditions, under a layer of molten slag for instance, the free portions of certain grains should assume their true external crystalline forms. It is possibly what has happened in some of the following instances.

According to Hausmann, as quoted by Fuchs,† the refined and ductile iron, formed in blast furnaces,‡ frequently exhibits some octahedral crystals. These crystals are met with more often in welding furnaces, when the blooms are overheated and burnt.

Woehler observed some cubic cleavages in a bar of forged iron which was part of the grate of a furnace used for melting silver.§ It is not, however, sufficiently certain that the temperature of the bar, cooled by the draught of air, was sufficiently

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* *Journal Iron and Steel Institute,* 1885, I, page 188.

† *Loc. cit.*

‡ The blast furnaces referred to here must be *Massofen* or *Wolfsofen,* as from the words *ductiles Eisen, Frischeisen,* it cannot be inferred that the metal described was cast iron.

§ *Annales des Mines,* 1883, page 360.

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high to justify the inference that its cleavages were due to the crystallization of iron in the *Gamma*-state?

Cornuel* "took from the interior of a puddling furnace of Cyrey-sur-Blaize a piece of crystallized iron, having a cubic cleavage, as can be seen from the small steps on the largest face of the fracture."

Gurlt† described a remarkable sample of burnt iron from the collection of the Direction of Mines of Berlin, found in a welding furnace in Silesia. The specimen is entirly covered with crystals having cubic cleavages and composed of a combination of cubes, octahedra and rhomboidal dodecahedra. The most frequent forms are the cube and the octahedron, less often the cube modified by the dodecahedron. The size of the crystals reaches one and a half inch; they generally have concave surfaces and are covered with a thin film of magnetic oxide; they do not contain any carbon and only traces of silicon; they are forgeable and very soft. Mr. Heyn‡ found some cubic cleavages in a furnace salamander containing very little carbon, since a microscopical analysis did not reveal the presence of any pearlyte; the percentage of phosphorus is not given.

Myself, I examined a piece of Swedish iron which had been partially melted and slowly cooled. A section etched with dilute nitric acid shows that the iron is arranged in parallel bands, and that these bands often cross each other, forming equilateral triangles (Fig. 31). The magnification is 16 diameters. These forms belong undoubtedly to the octahedron.

Summing up these observations concerning the structure of iron agree, as a whole, even after discarding those which are doubtful, with the observations recorded in the case of steel and cast iron. They all tend to prove that *Gamma*-iron crystallizes in the cubic system, assuming preferably the shape of octahedra more or less distant from its perfect form.

B. Structure of Quenched Carbon Steel. — It is natural to suppose that if a piece of carbon steel is rapidly cooled from a temperature exceeding that of the transformation of *Gamma*-iron into *Beta*-iron, the various transformations remain finally incom-

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* *Comptes Rendus,* Vol. XXXV, page 1852.

† *Uebersicht der pyrogeneten künstlichen Mineralien,* Freiberg, 1857.

‡ *Mitteilungen aus den Kön. tech. Versuchsanstalten,* 1898, page 311.

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Fig. 31

Fig. 33

Fig. 32

Fig. 34

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plete at the ordinary temperature. Even if they were complete, the structure corresponding to the *Gamma*-state, which existed at the instant of quenching, may be retained through pseudomorphism, for lack of time, if the transformation of the structure be slower than the molecular transformations. It is, therefore, possible that the crystalline forms existing at the quenching temperature are retained after complete cooling and that the structure of quenched steel will throw some light upon the structure of *Gamma*-iron. It is, however, only a possibility, and therefore, the observed facts can only be used under reservation and in the light of information.

Carbon steels may be divided into hyper- and hypo-eutectic alloys, according to their carbon content (the former if they contain over 0.90 per cent of carbon, the latter if they contain less than 0.90 per cent).

a. *Hyper-eutectic Steels.* — If steel be quenched in ice water at a sufficiently high temperature (1050° C. or more) to prevent the presence of cementite in the metal, the result is a mixture to which I have already referred, made up of austenite and of saturated martensite (the hardenite of Prof. Howe). In austenite at least, the iron has been retained in the *Gamma*-state, since this constituent undergoes the transformation when immersed in liquid air, like certain non-magnetic alloys of iron and nickel or of iron and manganese, with an increase of volume and of hardness. In certain regions (Fig. 32 represents a cemented steel containing about 1.50 per cent of carbon; the polished section was left 15 minutes in distilled water; the magnification is 250 diameters), the dark bands of martensite cross each other at angles of 90° and 45°; in other portions, and this happens more frequently (Fig. 33 shows the same steel etched with dilute hydrochloric acid while being connected with the positive pole of a Grenet battery, the magnification is 250 diameters), two systems of parallel lines cut each other at no definite angles and seem to suggest macles.* Some triangles, equilateral or not, are also met, especially in samples poor in austenite (Fig. 34 shows a

* By comparing Fig. 32 and 33, it will be noted that the line of demarcation which is very sharp in the first figure, is confused in the second. It is because the latter was taken from a section cooled, after quenching in liquid air. Austenite was changed into martensite, or something similar, and the outlines of both constituents were blurred.

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cemented steel containing about 1.20 per cent of carbon quenched in ice water at about 1050°; the magnification is 250 diameters; the sample was polished in relief and shows the martensite light and in relief, while the austenite is depressed and appears dark).

When the cooling of hyper-eutectic steel is a little less sudden (quenching in tepid water or in oil, cooling of small bars in the air), an excess of cementite falls out of solution in the shape of numerous fine plates, which appear to follow the cleavages of

Fig. 35

Fig. 36

Gamma-iron, the iron being in the *Gamma*-state when they are deposited. Figs. 35 and 36 show the structure of a bar of cemented steel, 12 mm. in diameter, containing about 1.50 per cent of carbon, and allowed to cool in the air after having been withdrawn from the cementation box. The cross section was polished and etched with tincture of iodine, so that cementite remains white under vertical illumination on a background of pearlyte passing to sorbite; the magnification (250 diameters) is not sufficient to resolve the structure of pearlyte. The plates of

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cementite form quite frequently by their intersection angles of 90° and 45°, thus recalling the appearance of Fig. 32 (austenite-martensite); more frequently they do not form any definite angles, quite seldom angles of 60°.*

b. *Hypo-eutectic Steels.* — Hypo-eutectic steels, quenched in cold water from a temperature superior to that of the critical points, are made up solely of martensite. The characteristics of this constituent are now so well known that it will not be necessary to illustrate it here. It is composed of clusters of parallel needles oriented like the three sides of a triangle, equilateral or not; rectangular arrangement is also found, but more seldom.

In short, the crystallization of quenched steel, owing to the frequent occurrence in prepared sections of equilateral triangles and of angles of 90° and of 45°, appears to belong to the cube modified by the octahedron; the recorded indications, however, are not conclusive.

C. Structure of Nickel Steel. — It is well known that certain alloys of iron and nickel containing about 25 per cent of nickel and 0.60 per cent of carbon, do not exhibit any noticeable critical points during slow cooling, and are, consequently, non-magnetic at the ordinary temperature. Iron is therefore present in such steel as *Gamma*-iron, and nickel in the *Beta*-state.

The structure of non-magnetic alloys of iron and nickel may, therefore, normally be, at any temperature above 0, that of *Gamma*-iron. I found, moreover, that the structure remains the same after the return of magnetism, when the nickel content reaches from 25 to 99 per cent, or, at least, that the crystalline arrangements assumed at a high temperature are not effaced in these alloys by the molecular transformations, so that they may be utilized.

Fig. 37 shows an alloy containing 58.3 per cent of nickel, which was forged and then reheated to about 1300° C. The polished section was etched during 15 seconds with hydrochloric acid, diluted with 9 times its volume of water, while being connected with the positive pole of a Grenet battery. The illumination was vertical and the magnification 25 diameters. It has exactly the same appearance as that of brass, containing from 0

* The samples of cemented steel used in these experiments were especially prepared by Mr. Grobot, manager of the Assailly steel works.

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Fig. 37

Fig. 38

Fig. 44

Fig. 47

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to 35 per cent of zinc and reheated to a high temperature. Mr. Charpy,* who has studied the microstructure of these brasses, finds in them octahedra and numerous macles. By analogy such may also be the case here, although it would be hazardous to affirm it. Fig. 38 shows the details of Fig. 37 under a magnification of 150 diameters.

When the alloys are not reheated after being forged, the structure is of course finer, but it retains exactly the same characteristics, a little distorted, unfortunately, by the numerous minute cavities produced by the etching on the surfaces of these nickel steels, and which, under high magnification, resemble small ill-defined negative crystals. (See Fig. 39, which represents an alloy containing 29.07 per cent of nickel, 0.14 of carbon and 0.86 of manganese; the etching was the same as in the preceding case, and the magnification 600 diameters.) The attention of the reader is called to the similarity between these photographs of nickel steels and those of iron etched by Mr. Saniter at a red heat. (Figs. 42 and 43.)

D. Structure of Manganese Steel. — Manganese, like nickel, when present in certain proportions, causes the disappearance of the transformation points of iron. Non-magnetic manganese steels may also, therefore, throw some light upon the crystalline forms of *Gamma*-iron, which in these steels are probably the forms normal at all temperatures.

I examined a sample made by Mr. Hadfield, after it had been reheated and quenched at a yellow heat, of the following composition:

Carbon 1.05 per cent, Silicon 0.24 per cent, Sulphur 0.07 per cent, Phosphorus 0.10 per cent and Manganese 12.25 per cent.

The analysis was made in the laboratory of the Hecla Works.

The grains are partly crystalline and partly pseudo-crystalline, the former prevailing in the quenched bar. A simple polishing in relief makes them apparent through slight differences of level giving rise to various shades in the photomicrograph (see Fig. 40, which is magnified 250 diameters), and which result from the crystalline orientation of the grains with regard to the plane of the section. The structure of the regions where crystallization is the sharpest, resembles very much that of nickel

* *Bulletin Soc. d'Encouragement*, 1896, page 205, Figs. 25 to 30.

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Fig. 39

Fig. 42

Fig. 43

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steel containing over 25 per cent of nickel, and that of very soft steel etched at a red heat by Mr. Saniter. Such structure may be due, therefore, for reasons already stated, to octahedra.

If the systematical examination of the preparation be carried further, either in polishing it on a piece of parchment moistened with a solution containing 2 per cent of ammonium nitrate, or in etching it with nitric acid diluted four times with water, or again in immersing it for 30 seconds in a bath containing 10 per cent of ammonium chloride in water while it is connected to the positive pole of a Grenet battery, groups of fine parallel lines appear in the grains, having the same direction in the same grain but different directions in different grains. These various methods, however, are uncertain, and they all leave deep and numerous cavities on the polished surface. It is much preferable to employ another method, first described by Mr. Charpy,* and which yielded very interesting results in some recent experiments by Messrs. Ewing and Rosenhain.† It consists in subjecting a polished surface to partial permanent deformation by striking the adjacent faces with a hammer; fine parallel lines are immediately seen, clearly defined; two or three systems of lines are seen in each grain (Fig. 41 shows a manganese steel reheated, under a magnification of 250 diameters). These lines evidently represent cleavages, but the angles of intersection are not constant, since the various grains are indifferently oriented with regard to the plane of the section.

The similarity between the structure of manganese steel and that of nickel steel is what we should remember.

E. Etchings at a Red Heat. — A last method of studying the crystalline forms of *Gamma*-iron consists in etching a polished section in the range of temperature in which this allotropic variety is stable.

The attacks at a red heat do not suppress the difficulties met with in interpreting the appearance of cold etchings, and they have their own sources of error.

1. The metal which is being operated upon has passed at least once, during its cooling from the molten condition, by the

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* *Génie Civil*, Dec. 26, 1896, page 117, Fig. 1.

† *Proc. Roy. Soc.*, May 25, 1899, page 85. *The Metallographist*, April 1900.

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Fig. 40

Fig. 41

Fig. 45

Fig. 46

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the three states *Gamma, Beta* and *Alpha;* during the heating preceding the etching it passes back from the *Alpha* to the *Beta* and then to the *Gamma*-state; some of these previous conditions may leave some traces of structural arrangement which may appear, after etching, superposed to the actual structure, and which may even be prevailing. At all events the results will have to be discussed critically.

2. If the steel be heated in the etching bath itself, then the etching will take place at a variable temperature, at least during the first period, while the metal is undergoing its transformations and the structure its corresponding transformations.

3. If the iron and the carbon are not acted upon with the same speed, the superficial composition will change during the etching, and the observed structure, supposing that it corresponds to the final composition, will not be that of the initial metal.

These objections, however, are not fatal to the method, which appears to be at least worthy of attention. It was proposed and applied by Mr. Saniter,* who selected, for his etching reagent, melted chloride of calcium maintained at a temperature of about 900° C. His results are certainly interesting. They are summed up below, and I have placed in opposition the results which I obtained with the same method, comparing also my conclusions with his.

MR. SANITER'S EXPERIMENTS.

a. When a piece of hyper-eutectic steel is etched at about 900 in melted chloride of calcium, the free cementite is burnt first, leaving dark veins dug in much more deeply than the mass, and this action continues until the bath contains a certain quantity of oxide of iron in suspension; the carbon of the martensite is then eliminated without any oxidation of the iron; finally the oxidation of the iron is resumed vigorously after the carbon is burnt.

To justify these conclusions Mr. Saniter immersed in the bath, dur-

MR. OSMOND'S EXPERIMENTS.

a. In a steel containing 1.00 per cent of carbon, the free cementite which may be present in small quantity, at the ordinary temperature, could not be acted upon first at 900, because it no longer exists at that temperature. In a steel with 1.8 per cent of carbon, some free cementite exists at 900, but it is not likely to be first acted upon, since it is a definite exothermic compound, which, in most known circumstances, offers more resistance to the etching action than the dissolved carbon.

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* *Journal Iron and Steel Institute,* 1897, II, page 115, and 1898, I, page 206. *The Metallographist,* Vol. I, 1898, page 72 and 251.

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ing various lengths of time, some drillings of two steels containing respectively 1.8 and 1.0 per cent of carbon; the carbon remaining was then determined and the residue weighed.

The results are shown below:

Temperature Seconds	Carbon % before etching	Carbon % after etching	Carbon dissolved %	Iron dissolved %
60	1.8	0.52	1.33	9.72
120	1.8	0.09	1.72	13.31
180	1.8	0.09	1.72	19.00
30	1.0	0.90	0.16	6.29
60	1.0	0.68	0.37	6.58
90	1.0	0.51	0.53	7.42

b. Mr. Saniter treated, by his method, a series of steels of various degrees of hardness, immersing them in melted calcium chloride during 15 seconds, after they had reached

The experiment confirms, as a matter of fact, the theoretical deductions.

A steel plate cemented by the Demenge process and forged, in which the carbon content varied continuously from 1.60 to about 0.30 per cent, was immersed and kept in motion in a bath of calcium chloride at about 900°, a first time during 32 seconds, a second time during 48 seconds, from the beginning of the immersion. The plate was quenched after each immersion. After the second treatment, the free cementite was found white and brilliant in the most highly carburetted regions, the balance being oxidized. (See Fig. 44, magnified 150 diameters.) As it was anticipated, therefore, free cementite, if any remain at the etching temperature, is not the first constituent to be burnt; the carbon of the martensite is the first one to disappear, that of the cementite being burnt only after it has been diffused. There is no ground for making a distinction between the two carbons.

On the other hand, Mr. Saniter's table shows that the carbon burns more rapidly than the iron. The figures of the table, however, do not give an exact idea of the facts, because the analysis of the drillings yield an average percentage, while the decarburization necessarily proceeds from the surface to the inside, the diffusion of the carbon not being sufficiently rapid to instantaneously make up for the losses.

b. A plate of open hearth steel, forged and containing 0.125 per cent of carbon, was etched in melted calcium chloride at about 960°, seven successive times, the immersions

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the temperature of the bath. As it is not otherwise stated, I believe his steels were forged.

His results were as follows:

Hyper-eutectic steels containing from 1.8 to 1.0 per cent of carbon: dark network the representing burnt cementite, the balance showing what the author calls *acicular* structure, and which he compares, rightly, I believe, to that of martensite (Fig. 42, magnification 200 diameters).

Steel with 0.60 per cent of carbon: acicular structure very pronounced.

Steel with 0.43 per cent of carbon: acicular structure feebly marked.

Steel with 0.10 per cent of carbon: no acicular structure; division into grains which is found in all the samples.

lasting respectively 24, 28, 28, 32, 48, 64 and 130 seconds. The metal was held with a pair of tongs, which retarded its heating. After the first immersion it was incipient red; after the three next immersions, dark red; after the fifth, cherry red; after the sixth, light red; and after the seventh immersion, yellow. The plate was quenched after each immersion. After the first five immersions the structure was found to remain indistinct, the etching being insufficient, and, moreover, the sample not having reached the proper temperature. After the sixth treatment the metal was found to consist of grains divided by a network of joints; the ferrite appeared amorphous, and the preparation was exactly similar to that obtained by Mr. Saniter with his steel containing 0.10 per cent of carbon. After the seventh immersion an acicular structure was obtained, a little more crystalline possibly, but otherwise analogous to that which Mr. Saniter considers characteristic of hard steel (compare Figs. 45 and 46, which are magnified 600 diameters, with Fig. 42). Both photomicrographs (Figs. 45 and 46) were taken at the same spot; the first one a little above, the second a little beyond the focal point; the former shows better the joints of the grains, the latter the internal structure.

The sample of steel whose carbon content varied uniformly from 1.60 to 0.35 per cent, and which had been subjected to two immersions, respectively of 32 and 48 seconds, as described above, was now immersed a third time during 72 seconds; this time, as noted by Mr. Saniter, heavy black

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markings indicate the place occupied by the network of cementite. This net work, however, is broken and about to disappear (see Fig. 47, magnified 150 diameters); the rest of the structure is indistinct, with the exception of the intergranular network in the soft region of the plate. Finally, after a fourth immersion lasting 92 seconds, the black network corresponding to the cementite disappears more and more, and the acicular structure may be seen everywhere, and better defined towards the soft extremity of the bar.

c. Mr. Saniter argues that if the acicular structure belongs to *Gamma*-iron, it should be detected in all his samples, and as it is, on the contrary, according to his observations, dependent upon the carbon content, he infers that it is probably characteristic of a definite carbide of iron, resulting from the quenching treatment.

c. Mr. Saniter's experiments had shown that the carbon content of the outside of the plates, which regulate the final structure, is certainly inferior to the initial percentage.

My experiments prove that the acicular structure is independent of the initial carbon content, since it may be obtained while the carbon varies from 0.25 to 1.60 per cent.

The acicular structure, therefore, does not belong to a definite carbide of iron.

Of course, in the conditions of Mr. Saniter's experiments, i.e., the temperature and the length of immersion, being constant, the appearance of the surfaces may depend upon the carbon content:

1. Because the stage of the crystallization of the iron at a constant temperature is a function of the initial carbon content;

2. Because the final carbon content, the length of the immersion remaining the same, depends upon the initial carbon content.

The acicular structure, however, can only be identified with the factor remaining constant throughout the variations of the carbon

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d. Previous experiments had been conducted with some samples in the *cast condition or thoroughly annealed.* The etchings lasted a little longer (25 seconds instead of 15). The results are given below.

Pure Iron. — Distinct indications of crystallization; a nearly perfect parallelogram was detected as well as a triangle and some angles none of which were right (see Fig. 43, magnified 1000 diameters). Mr. Saniter calls this structure *allotropic,* and thinks, that it might belong to the hexagonal system.

Steel with 0.10 per cent Carbon. — The structure is made up of small grains (supposed to be carburized) and of large grains (supposed not to be carburized). Indication everywhere of allotropic structure.

Steel with 0.185 per cent Carbon. — The small grains (supposed to be carburized) are prevailing, but appear amorphous; the structure of the large grains appear to be allotropic.

Steel with 1.00 per cent of Carbon. — Acicular structure as stated above.

e. Mr. Saniter, therefore, distinguishes in steel at about 900° the three following structures:

Allotropic in pure iron;

Amorphous in medium-hard steel;

Acicular in hard steel.

He infers from the above that while iron undergoes a molecular change in pure iron (A_3), the change has a tendency to disappear as the percentage of carbon increases, and does not take place in hard steel. Carbon is the enemy of allotropic iron.

content, before and after the etching, that is, with *Gamma*-iron.

d. In the second series of Mr. Saniter's experiments, the crystalline appearances, yielded by pure iron and very soft steel, represent probably the structure assumed *during solidification or during the thorough annealing* of the steels experimented upon, and not the structure at the time of etching. Similar steels similarly prepared would have yielded by the ordinary cold etching, as seen above, appearances analogous to those resulting from etching at a red heat, and it is natural that the reheating necessary to thus etch it, should not completely efface the preëxisting structure.

These characters, moreover, are altogether similar to those found in the case of manganese and nickel steel, and on that account I agree with Mr. Saniter in attributing them to *Gamma*-iron. I, however, see no reason why they should belong to the hexagonal system.

e. Just as the allotropic structure belongs to *Gamma*-iron so does the acicular structure. There is no essential difference between the two structures: the second is the germ of the first; the first is the limit of the second when crystallization is encouraged through a sufficient rise of the temperature. They are both characteristic of *Gamma*-iron.

As for the amorphous structure, it does not exist, strictly speaking; the etching methods may, however, be unable to reveal a very fine crystallization. It is known, moreover, that carbon does not remove the point A_3 (transformation of

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Gamma into *Beta*-iron, or inversely), no more than it attenuates it. The specific signs of the point A_3, such, for instance, as the abnormal dilatation during cooling, are found in irons, steels and cast irons; such is also the case with the structure. Carbon, therefore, is not the enemy of *Gamma*-iron, and as it retains iron in that state, during cooling, below the normal temperature of the transformation, it would, on the contrary, be more accurate to say, following the metaphor, that carbon is the friend of *Gamma*-iron.

To sum up, concerning the question under consideration, the experiments by Mr. Saniter's method have shown that in the range of stability of *Gamma*-iron the structure of carburized iron is usually that of martensite and probably, near the melting-point, that of non-magnetic manganese or nickel steel, the latter being the limit of the former, as the conditions become more and more favorable for a perfect crystallization.

F. CONCLUSIONS. — Of the five sources of information to which we have turned, only the first one, i.e. the examination of the crystals found in the cavities (pipes) of cast pieces, yields the crystals found in the cavities (pipes) of cast pieces, yield crystalline forms freely developed, and the study of these forms appears to classify them, undoubtedly, with the cubic system, octahedral modification, with probable octahedral or cubic cleavages.

The four other sources have given indications which are not conclusive as such, but which are concordant or, at any rate, consistent with the conclusions derived from the first source.

(To be concluded in the next issue of "The Metallographist.")

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PRACTICAL METALLOGRAPHY.*

By J. E. STEAD.

IT is many years since Dr. Sorby, of Sheffield, applied the microscope to the study of the structure of steel. For a long time after the publication of original papers, nobody took much interest in that method of research. During the last few years, however, the interest has revived, and the microscope is now regarded as almost indispensable in a large number of the metallurgical laboratories of the world. The interest is increasing, and I am very often asked to give advice as to what apparatus is required in order to prepare metal sections for microscopic examinations. It occurred to me that if I described the methods which have been found most convenient and useful in my own practice, it would be of some assistance to others who wished to take up this most interesting study. The fact that this meeting is so largely attended, fully justifies the step I have taken. It is proposed to describe and demonstrate practically before you how I cut, polish, etch, and photograph the metal sections, and show you by means of diagrams, etc., the apparatus I have used with success.

Practical metallography may be studied under the following sections:

1st — Preliminary preparations of the specimens.
2nd — Methods of polishing.
3rd — Etching the specimens.
4th — Mounting the specimens.
5th — Necessary microscope accessories.
6th — Photographing the developed structures.
7th — Lists of apparatus.

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* *Proceedings Cleveland Institution of Engineers,* February 26, 1900. This meeting was somewhat novel in its character. It was not, as usual, for the purpose of hearing a paper read with illustrations, but of listening to an address on a new subject of investigation from one of the original investigators with a practical exposition of his manner of investigation, as an inducement to other members to join him in the study. The room was studded with microscopes, through which could be seen the results of the lecturer's work and before him on a bench on the platform, were the instruments with which he had done the work. His object that night was to show practically how it was done.

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No. 1.—Preliminary Preparation of Specimens.

When time is no object and labor of little account, it is advisable to obtain all the metal sections in the form of little plates about ⅜ inch to ½ inch in diameter and $^1/_{10}$ of an inch in thickness. This can easily be managed if the metals are soft enough to be sawn, filed or turned into shape, but it is very difficult, unless a diamond cutter is available, to obtain harder metals of such dimensions; I might say it is almost impossible. Fortunately it is not really necessary, for as only one plane surface is required for examination it does not matter how irregular the other portion is.

This being so, the only difficulty is to readily mount the object so as to leave the polished surface horizontal with the glass mounting slide. This has been overcome in a very simple manner and will be described presently.*

In preparing pieces of white pig iron and other hard brittle metals or alloys it is only necessary to take a chip or fragment, preferably not more than ½ inch in thickness, with one comparatively smooth face. This face is ground down on an emery wheel till it is flat, then finished in the manner to be presently described.

It saves much time, and an emery wheel can be dispensed with, if the surface to be polished does not exceed ¼ inch in diameter. Emery paper will be capable of grinding to flatness such a small area.

When sections of wires are to be examined it is advisable to take a small piece of high carbon steel ¼ inch round by $^1/_{10}$ inch in thickness and drill a hole through the center just large enough for the wire to fit it. The wire is soldered at the back to its support, care being taken to keep the face with the wire protruding cool by placing it on a metal block when soldering. The wire can be cut off close to the back of the support. The face of the piece is then ground and polished. The end of the wire remains quite flat after polishing.

In supporting sections of thin plates pieces of steel about ½ inch square and ¼ inch deep are cut two-thirds through with a hack saw. The thin plate is inserted into the cut, and the sides

* See foot-note, page 231. Ed.

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of the supporting steel are pressed against the specimen by pressure in a vice, or by a sharp blow from a hammer. If necessary, three or four pieces can be mounted in the same little block. When secured in this way the specimens are ready for polishing.

In examining the worn surfaces of rails or tires which are often covered with microscopic cracks, it is clear that such surfaces must not be ground down to obtain a perfect polish, for by doing so the area in which the cracks are situated will undoubtedly be removed. It is therefore advisable to polish with the finest emery paper, No. oooo, and to finish on rouged leather. This treatment will leave a very rough surface, pitted with rust marks, but quite clean enough for the purpose. Probably the cracks will be visible under a strong lens without any previous treatment with acid, and if not by a lens, by a low power objective under the microscope. If nothing can be detected after polishing alone the specimen must then be allowed to remain for thirty seconds in a solution consisting of one part of nitric acid in 100 of water, and after washing well with clean water it is soaked in lime water, washed again with water, and finally polished up on rouged chamois leather. It is then examined, and the cracks will most probably be clearly seen; if they are not, again immerse in the acid, and proceed as before. If no cracks appear, soak in a 5 per cent solution of nitric acid (5 c.c. acid of 1.42 specific gravity, and 95 c.c. water) for one minute, and wash as before and examine. If now no cracks are visible they may be considered to be absent. In the preparation of a vertical section, after having cut out a horizontal slice about ¼ inch in thickness, and ¼ inch in width, and ¾ inch in length; cut it vertically into two halves, so as to make two pieces each ¾ inch long, $^{3}/_{32}$ inch wide, and ¼ inch thick. Rub the two head surfaces on the finest emery paper to remove excrescences produced by the saw; then place them against each other, and screw them in that position in a vice, taking care to have the two pieces exactly parallel. Now smoothly file the upper surface projecting from the vice, and dust with resin powder, and with the soldering iron drop a piece of very fusible solder on to the filed surface, and apply the soldering iron until it unites with the steel. This should be done so rapidly as not to cause the steel to be materially heated. Now turn the soldered side down and secure in vice, file smooth, remove from the vice, and grind on emery, and finish on cloth

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and rouged parchment or leather. You will now have a specimen quite flat across the center part where the two head surfaces rest one against the other, and you will have two vertical sections, the fine line in the center, of course, being the division between them.

The object of placing the two surfaces together in this way is to avoid the slight rounding or curving at the parts where it is most important they should be perfectly flat. If the precaution is not taken when polishing on soft beds, the edges get worn down, and are then unsuitable for examination, especially under high powers.

No. 2. — Polishing Metal Sections for the Microscope.

Dr. Sorby, who is the pioneer in micro-metallography, used such simple, yet effective methods for preparing metal sections that anyone following in his footsteps can, at a cost of two or three shillings, equip himself with all the material necessary, and with a little pains produce excellent sections.*

Those who have seen Dr. Sorby's preparations admit that none better have been prepared since. Much time is taken in perfecting each specimen, but the result is perfection.

Prof. Arnold's process is similar to Dr. Sorby's, the details only being modified. Neither gentleman uses mechanical means for effecting his object.*

Mechanical Devices for Polishing.

Mr. Osmond follows closely the method of Dr. Sorby,* but the final polishing is accelerated by the use of a kind of lapidary wheel, upon which he secures the polishing cloth and powders. Mr. Osmond, however, has made a very important departure from the original methods of polishing by developing the structure of steel, etc., by what he calls "the polish attack." This will be described presently.

Prof. Martens kindly sent me a pen and ink sketch of an apparatus he employed for polishing specimens, and Mr. Harrison has made a drawing of it, which is on the table before you. (Fig. 1.)

---

* See *Journal of Iron and Steel Institute,* No. I. 1894.

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You will see that it is designed to polish a series of specimens at the same time. The block carrying the thin sections of metals is caused to traverse the face of the grinding and polishing bed of the power-driven wheel.

I have had no experience in polishing on this machine, but

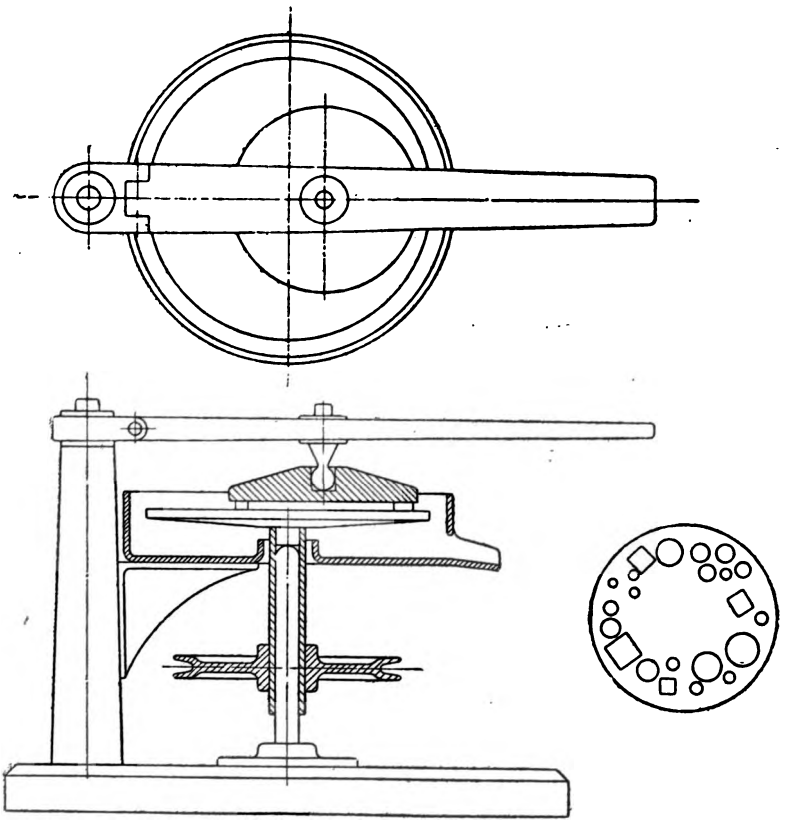

Fig. 1

there can be no doubt that, provided the metals cemented to the block are relatively of the same hardness, excellent work will result. The fine work of Prof. Martens is proof of this. During the repeated trials I have made to produce a really serviceable mechanical polishing machine, at first I endeavored to polish many specimens at the same time by mounting on a block, but

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that was soon abandoned, as, in practice, it was a rare thing to have many metals of the same hardness to polish at once. Each

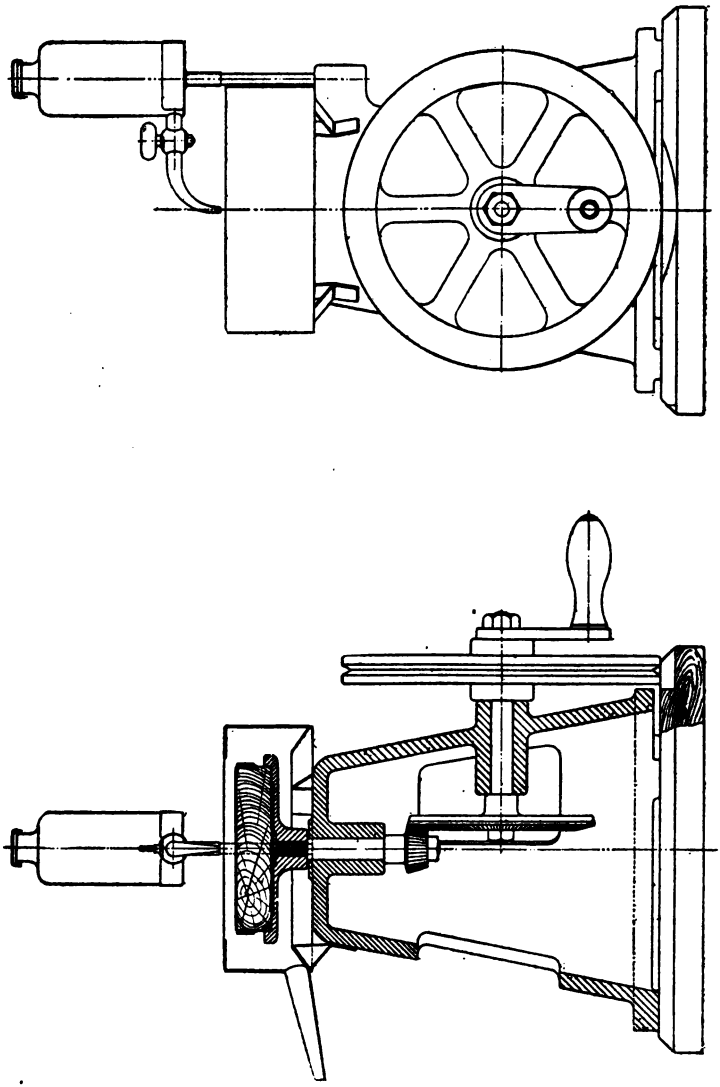

Fig. 2

specimen requires its own special treatment, and it was soon found that they could not be polished alike.

In the apparatus I first designed, made by Messrs. Carling

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& Son, Middlesborough, a mechanical holder was attached, upon which the single specimens were either cemented or soldered, and this was made to traverse the polishing bed on the revolving wheel, but it was soon found that the more simple plan of holding the specimen with the first finger of the left hand, while turning the wheel with the right, enabled one to save much time, and regulate the pressure more correctly than with the swinging arm; the latter was therefore abandoned.

The apparatus has been improved, and is now, I believe, as simple and efficient as a hand machine can be made; it is really a combination of the designs of Mr. Osmond, Prof. Arnold and Prof. Martens.

The drawings prepared by Mr. Harrison will be understood without much explanation. (Fig. 2.)

1. There is the horizontal revolving wheel driven by the hand at the right of the machine.

2. Conical blocks with rings for securing in position the emery papers, etc. There are several of these blocks for carrying the different polishers. They are simply dropped into the recessed wheel and are carried round by friction.

3. A cooper can for holding either water or ammonium nitrate solution and a tap for allowing the solution to drop on the center of the polishing blocks.

4. A shield for catching the fluid which is thrown off the wheel when it is at work.

5. Cases for the blocks to prevent dust falling when not in use.

Messrs. Carling & Son assisted me greatly in designing the machine first described. They have now designed a new machine on the same principle as the first described, but it is lighter, can be driven at greater speed, and will work silently, as the small pinion wheel is to be made of compressed leather.

I pointed out to Mr. Carling that the blocks might be, with advantage, reduced in size if the speed is increased. The smaller the area on which grit or dust can fall, the more easy it will be to keep clean. Acting on this suggestion the new machine will have much smaller blocks. (Fig. 3.)

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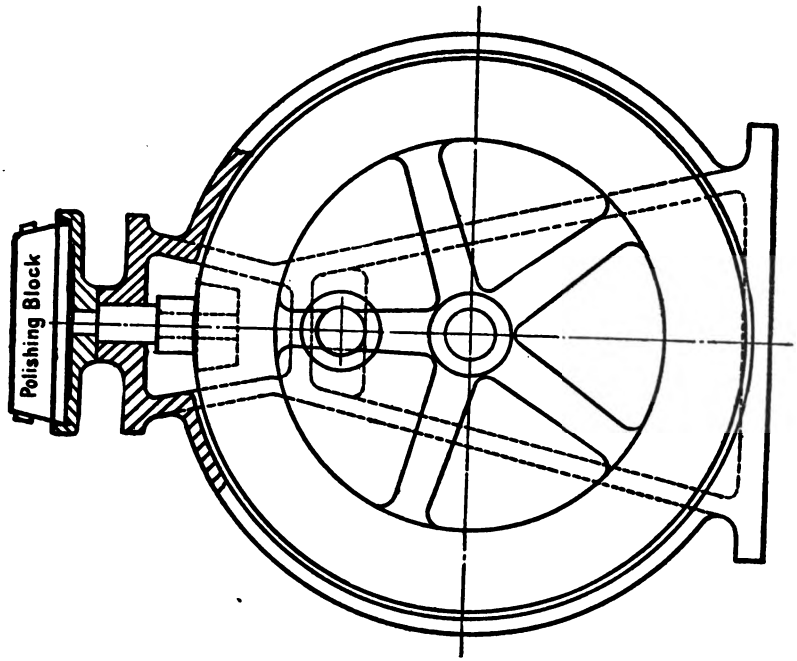
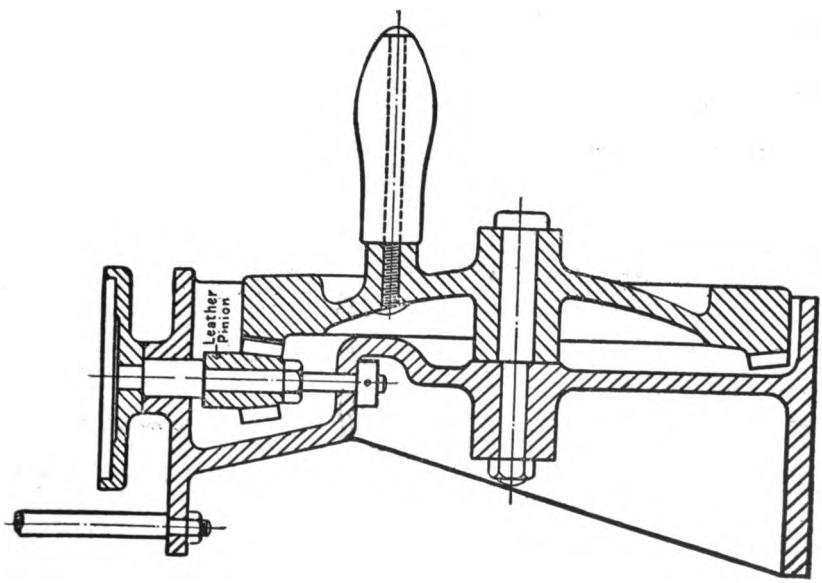


Fig. 3



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Practical Demonstration.

It is two years since I promised to give to this Institution a practical demonstration of the methods of polishing and etching and photographing metal sections, and I am here to-night to redeem that promise. I shall take for my trial a ⅜" bar of steel, containing about 0.40 per cent carbon.

1. The bar I place in a vice and file one end quite flat with a smooth file.

2. It is now fixed into a second vice attached to a hack saw and a section is cut an eighth of an inch in section.

3.* A No. 0 emery-paper block is placed on the machine, and with the first finger of the left hand the specimen is pressed on the block while the wheel is being revolved with the right hand. The specimen is kept in one position till all the file marks have disappeared, and the emery scratches are all in one direction. The specimen is now turned 90°, and the pressure having been reduced the wheel is again made to revolve till again the scratches are in one direction, but at right angles to those first made.

4. A second block carrying finer emery paper No. 00 is now placed on the wheel, and the same treatment given to the section as just described under No. 3.

5. A still finer block is now used, No. 000, and the same treatment repeated.

6. The next block is covered with smooth black cloth, and upon its surface is rubbed about one grain of Diamontine powder used by jewellers for polishing steel. Water is allowed to drop on the block from the copper bottle, and the wheel made to revolve. The specimen is pressed on the block and made to traverse across it so as to produce even polishing.

It is examined from time to time in a good light till all the fine emery scratches are removed. The polish at this stage is sufficiently good for most practical purposes, but if a photograph is required it must be passed over a finer block.

7. The final block, suitable for this steel section, consists of chamois leather and fine gold rouge. The rouge is well rubbed in, and then the wheel is caused to revolve rapidly, whilst the

* The emery papers are those used by steel engravers for polishing steel plates. They are made in France and can be obtained through any good ironmonger.

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surface is pressed with the flat edge of a thin strip of steel. This loosens the coarser particles of rouge, and they are thrown off the block, and none but the finest parts remain. The specimen is now placed on this block, and very gentle pressure applied and the wheel revolved till, on examination, the surface appears to have slightly lost its brilliancy. If the polishing is carried too far, the fine surface is destroyed, and it becomes very uneven and dull.

The polishing has been stopped at the right moment and it is ready for etching.

8. For finer structures the dry block with rouge is not good enough, and No. 7 is replaced either by parchment or by fine kid leather, the latter being recommended by Prof. Arnold.

In either case the block is first soaked in water and then the rouge is well rubbed into the surface; the rubbing is continued under a stream of running water which effectually removes all but the finest articles of the rouge (this is Osmond's excellent plan). The block prepared in this way is placed on the wheel, and the section of metal pressed upon the surface. Water in just sufficient quantity is allowed to drop on the block so as to keep it quite wet but not sufficient to cause it to flow off.

It takes longer to polish on wet blocks than on dry, but the results are much superior.

9. In making the "polish attack" of Osmond, parchment is used on the block, but instead of rouge and water, sulphate of lime (prepared by adding a boiling solution of sulphate of ammonia to a hot solution of calcium chloride and thoroughly washing the precipitate) and liquorice root solution are used.

Treated in this way many specimens of steels and other metals have their structures developed most perfectly without any further treatment.

Cementite, pearlyte, martensite, troostite and what Osmond calls sorbite, are clearly developed by this method.

In the January, 1900, issue of *The Metallographist* is a translation of a paper by F. Osmond and G. Cartaud, on "New Reagents for the Micrographic Study of Carburized Irons," in which is described a valuable substitute for infusion of liquorice root for making the "polish attack."

In concluding this section, you have observed that the polishing and etching of this sample has not taken more than fifteen

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minutes, and I can assure you that when the operator is not obliged to talk as well as work, the time need not exceed ten minutes. As a matter of fact, the average time taken in polishing the thousands of sections I have prepared has not exceeded ten minutes. A novice, at first, will probably take half-an-hour or more, but in time, when experience teaches how to graduate the pressure in polishing, the shorter period will suffice.

The cutting of suitable sections from a large mass of steel may take a whole day; such preliminary work must be done in a machine shop.

The following facts should, however, be noted:

a. The softer the metals the lighter must be the pressure, and they must never be polished on dry blocks. Lead is the most difficult of all metals to polish, and it is only by the slightest friction long continued that any good polish can be obtained.

b. Hardened steels and white irons require much greater pressure and are easily polished.

No. 3. — Etching the Specimens of Iron and Steel.

1. The "polish attack" of Osmond has already been described. It takes the first place and gives the most perfect results in very many metals and alloys.

2. The second method of etching steel is by a tincture of iodine of the following strength:

Iodine	1.25 grms.	100 c.c.
Iodide of potassium . .	1.25 "	
Water	1.25 "	

Alcohol to make up 100 c.c.

This is the reagent used by Osmond.

A second weaker tincture, one-fourth the above strength, is very useful.

The polished specimen is placed on a plate, and one drop per square centimetre is allowed to cover it. It is left there until the iodine color is gone. The specimen is washed in water, then in alcohol, and finally dried in a blast of hot air. It is mounted and examined. The iodine treatment is repeated if the first application has not sufficiently developed the structure.

Some metallographists advise the wiping of the specimen

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with a clean linen cloth instead of drying, but this occasionally removes colored deposits which are sometimes a most important indication. After drying by hot air and examining under the microscope, the specimen may be afterwards wetted and then dried with the cloth and reëxamined.

3. Etching with dilute nitric acid is often recommended; but it is not so safe for steels as tincture of iodine, but in the absence of much phosphorus and arsenic it gives good results.

The specimen is immersed in a vessel containing a 0.10 per cent nitric acid (1.42 sp. gr.) in water, and is retained there until the structure is developed sufficiently clear. The progress of the etching should be watched through a strong magnifying glass. After removing from the acid it should be well washed by running water, immersed in lime water, rewashed in water, then in alcohol, and finally dried with hot air. Fletcher's hot blast blowpipe answers well for this purpose.

4. Osmond recommends a 20 per cent solution of nitric acid for rapidly developing the crystalline or granular structure of steels.

The specimen is immersed in the acid from one to ten seconds, and for even longer periods, then removed, washed as before and examined. This I have found to be very useful when deep etching is required.*

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* Mr. Stead does not mention the treatment with concentrated nitric acid as described by the writer on several occasions. As it has been found by him to generally give better results than the etching with dilute acid, a previous description of the method may perhaps be reproduced here with some profit to the reader:

"The unreliableness and other shortcomings of the treatment with dilute acid led the writer long ago to discard it and to adopt the following method:

"The polished sample, suitably held, is dipped into concentrated nitric acid (I. 42 sp. gr.), which, on account of the passivity of iron, has little or no action on the polished surface. The specimen is then placed under an abundant stream of running water and the acid is quickly and completely washed off. As soon as the layer of concentrated acid which covers the surface is diluted by the running water, it attacks the steel, at first vigorously, but for such a very short time (since the water soon removes all trace of acid) that there is no danger of etching too deeply.

"Such treatment develops the structure sharply and clearly, the etching being of uniform intensity all over the surface and free from the objectionable colored film and from the unlike appearance of different

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5. Prof. Behrens and Prof. Martens have used, with great success, the simple process of heating the steel and other specimens till they take oxidation tints. This system may be described as a "*heat tinting*" one, as it can scarcely be called etching.

I have latterly used the method with most satisfactory results, and have found that it is the only method by which phosphides and carbides of iron can be distinguished from one another, when associated in iron and steels.

In preparing the specimen for heat tinting, the bright surface should be well rubbed with a clean piece of linen or chamois leather. Care must be taken to avoid any condensation of water on the surface; this is best avoided by warming the specimen to 90° or 100° C. before giving it a final vigorous rubbing with the linen or chamois leather, and before it cools it should at once be placed on the iron plate. A sheet of iron 6 inches square placed over a Bunsen burner is all that is required for heat tinting. The section is placed in the center, and the polished surface watched till the proper tint is obtained. Practical experience will soon enable one to find out the best tint for any particular metal or alloy It is best to heat gradually and examine periodically under the microscope, and stop when the structure appears to be most perfectly colored. After each heating the section should be placed in a dish containing mercury, so as to cool it rapidly and check further oxidation. If it is desired to photograph the heat-tinted object, it is most important in many cases to stop the heating when the object assumes a pale yellow tint, for although the contrasts to the eye are not so great, they are quite great enough for the sensitive photographic plate.

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parts of the field, caused by local actions of varying intensity, which are so troublesome and misleading in etchings with dilute acid.

"It is sometimes necessary to repeat the treatment in order to develop the structure to the proper depth, but more than two immersions are seldom required. The specimen is washed in water and alcohol and quickly dried with a soft cloth or under a blast jet." — Ed.

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No. 4.—Mounting the Specimens.*

When the specimens are quite flat, they may be mounted with Canada balsam, or wax, upon the glass slips. As it is rarely (unless the specimens have been prepared in the lathe) that the two sides are parallel, it is necessary to provide some means of mounting so as to have the polished surface perfectly horizontal with the glass slide on which it is mounted.

After trying many plans I find that the simplest method consists in placing the polished face of the section on a piece of plate glass, and placing over it a short brass cylinder accurately turned of such depth that the back of the specimen does not stand above the cylinder. A piece of plastic wax, used in Kindergarten schools for making models, is stuck upon the center of a glass microscope slide, and this is then pressed upon the section till the glass slide comes in contact with the brass ring. The specimen adheres to the wax, and the mounting is complete. It may be made more secure by dressing up the wax against the section and glass plate with a hot wire.

After examination, if it is desired to preserve the section, I prefer to cover it with a solution of paraffin wax dissolved in benzole. The benzole slowly evaporates, leaving a thin protective coating of wax. It will prevent the formation of rust, and when it is required for reëxamination, the wax is dissolved off by wiping it with a clean linen rag moistened with benzole. It is finally gently rubbed with a dry linen rag.

The brass cylinder rings can be made by any fitter by cutting up a brass tube 1″ internal diameter in a lathe, into lengths of "0.1, "0.15, "0.20, "0.25, "0.35, "0.40, "0.45, "0.50, "0.60. They must be accurately faced, after being cut. Messrs. Carling & Son have expressed themselves willing to make these rings at a cost of 5s. per set of 10, in a suitable case.

In the course of etching the specimen, it is inadvisable to have it mounted on the slide, as when mounted it is difficult to

* Mr. Stead is evidently not aware of the existence of a very simple device by which any specimen, however irregular in shape, may be immediately placed upon the stage of the microscope in the proper position to be examined, thus doing away altogether with the troublesome and unsatisfactory operation of mounting each sample upon a separate slide. These specimen holders greatly simplify the manipulations of the metal microscopist. — Ed.

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remove the water used in washing from the irregular under surface of the metal, and this, if left there, causes rusting.

To temporarily hold the specimens in position, I have used with success a glass slide on which is cemented a short length of brass tube. This is nearly filled with sea or silver sand.

The microscope being in a vertical position, the special slide, with its cylinder containing the sand, is placed on the stage. The "object" is placed face upward on the sand, and a piece of glass plate pressed upon it till it meets the walls of the brass cylinder. The glass is then removed, leaving the specimen bedded in the sand with its polished surface parallel with the bed of the stage. After preliminary examination the section can be removed with a pair of forceps, and further treated or, finally, be mounted for storage.

Prof. Arnold's method of mounting and marking the specimens is described in my paper on Methods of Preparing Polished Surfaces of Iron and Steel for Microscopic Examination" (*Journal of Iron and Steel Institute,* No. 1, 1894.)

When a cover-glass is admissible, it is possible to cover up the polished surface in the usual way with Canada balsam, and many of my specimens mounted four years ago in that way are still quite bright.

Mr. Saniter has informed me that he mounted all his specimens in this way, and they are certainly well-preserved.

Personally, I prefer to do without the cover-glasses, as when examining with low-power objectives under vertical illumination, the surface of the glass sends back reflected light, and this quite spoils or prevents clear definition. With oil-immersion objectives, the glass does not interfere.

No. 5.—Microscope Accessories.

There are many published treatises on the microscope, and students are referred to them for general information.

It is only necessary to describe here those parts of the microscope outfit which have been specially designed for illuminating opaque objects and for changing the object-glasses.

The systems for illumination may be divided into two main classes:

a. For illuminating by light thrown on the object by re-

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flectors fixed below the object-glass, only admissible with low-power objectives when there is ample distance between the object-glass and the object.

b. For illuminating objects when using higher power objectives than about ½-inch focal length, mirrors or prisms placed above the object-glass are necessary.

a. *Reflectors for Low-Power Objectives.*

The combination sold by Messrs. Beck & Co., known as the Sorby-Beck reflectors for oblique and vertical illumination, is the most useful yet designed. The following illustrations (Fig. 4) will show the construction of this piece of apparatus.

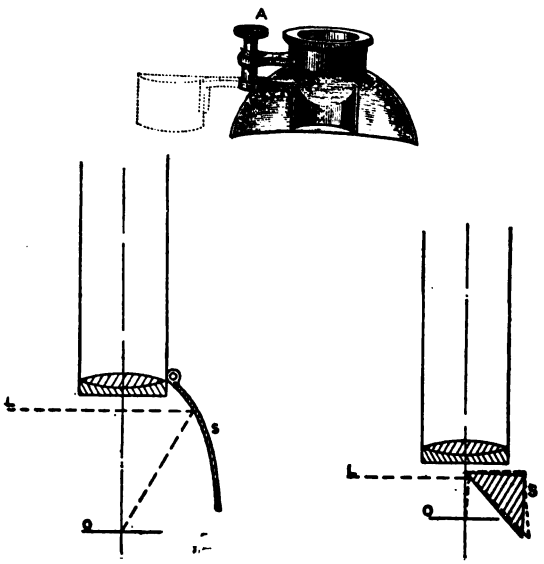

Fig. 4

When working with the parabolic reflector of Beck, the little silver mirror of Sorby, with a face at 45° to the horizontal, is thrown outside by turning the milled head A. The horizontal rays of light falling on the silvered parabola are reflected at various angles upon the object placed below, and if the object is a perfectly flat surface, the whole of the light will be thrown off, and none will enter the object-glass; hence, such a flat surface cannot be seen on looking through the microscope, — it appears quite black.

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The parabolic side reflector, on this account, is most useful in determining which part of a structure is horizontal, and which broken up.

It is useful also in defermining when one constituent is in relief, for as the light falls on one side of the projecting portion, it brightly illuminates it, leaving the other side in shade.

The vertical illuminating mirror of Sorby, when swung over the object, partly eclipses the object-glass, but the horizontal rays of light, falling on the mirror, reflect vertical rays upon the object, and these rays are again reflected into the microscope.

It is not, however, necessary to use this special apparatus when only vertical illumination is required. A piece of blackened

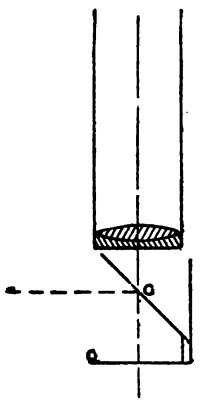

Fig. 5

cardboard fixed behind the object in a vertical position, and a cover-glass placed over the object at an angle of 45°, gives excellent illumination. The card and cover-glass can be temporarily attached to the object-glass slip with plastic wax. It only takes one minute to arrange (see Fig. 5).

The horizontal incident rays from the lamp are reflected downwards on the object, and thus brightly illuminates the specimen. The light from the object passes upwards through the reflector and into the object-glass and microscope.

b. *Reflectors for High-Power Objectives.*

The Beck illuminator for high power is similar to the last described, but it is placed inside of a special adapter which is screwed into the microscope tube above the object-glass. The

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light is admitted at the side of the adapter through an opening which is capable of adjustment by a suitable diaphragm (see Fig. 6).

The light falling on the cover-glass is reflected downwards through the object-glass on to the object. The rays from the object pass upwards again through the object-glass and the reflector to the eyepiece.

Prof. Mattens and others use a right-angled glass prism above the object-glass, adjustable by a rack and screw, so as to move it in or out in a horizontal direction. The light from the prism passes downwards in a vertical direction, illuminating the object most perfectly. Messrs. Nachet & Sons are makers of a most perfect illuminating device of this kind which, in my hands,

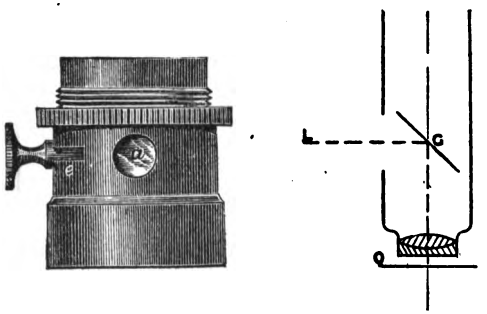

Fig. 6

has given most excellent results when photographing up to 500 diameters (see Fig. 7).

I am indebted to the Messrs. Nachet & Sons for the printing-block used for the above.

Object-Glass Slide-Changer.

It is constantly necessary in examining metal sections to view the structure under different object-glasses, commencing with a low-power, such as an inch objective fitted with a Sorby-Beck reflector; next, with a ¼-inch or ⅛-inch objective with the Nachet prism illuminator; and finally, with a ⅛-inch or $^1/_{16}$-inch oil-immersion objective with the Beck illuminator.

For ordinary microscopic work, when it is not necessary to have the somewhat cumbersome illuminating auxiliary apparatus

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attached to the object-glasses, the triple nose-piece is all that is required; but as the illuminators interfere with the free rotation of the nose-piece, it is not suitable for metallographic work.

Carl Zeiss, of Jena, has designed a much more useful changing apparatus, which is called by him an Object-Glass Slide-Changer.

Changing from one object-glass to another does not take more than two or three seconds.

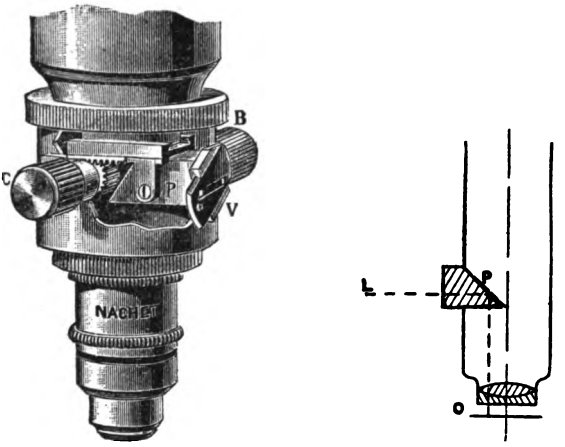

Fig. 7

Illumination.

It is absolutely necessary to have command of a brilliant light, more particularly when the metal objects have to be photographed.

If electricity is available, either an arc lamp or a forced incandescent light is the best; failing electricity an incandescent Welsbach gas lamp, or an acetylene jet will answer well. I have used with success the Welsbach lamp for all my work.

A bull's-eye condenser should be placed between the light and the reflector, so as to concentrate the rays as much as possible.

In many cases it is necessary to cut off the full force of the light by means of diaphragms or stops, especially when using the oil-immersion high-power objectives, and in all cases when the object has a brilliant reflecting surface.

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This is easily done by using the stops or regulators supplied with the Beck & Nachet reflectors.

The light may be more diffused by placing a piece of ground glass or thin tissue paper in front of the lamp.

It is only by experience and practice one can find how best to illuminate an object.

No. 6.—Photographing the Object.

Of cameras for photographing microscopic objects there are very many. Nearly all the manufacturers of microscopes also make cameras for this purpose. There are two systems of using these cameras. The first is that in which the microscope is turned over so that the tube is horizontal and the eyepiece is fitted into the front of the camera, which is also placed in a horizontal position. The second is that in which the microscope and camera are placed in a vertical position.

In the first case, the microscope must be fitted with a mechanical stage, and the objects must be securely mounted on the glass slides. The long camera monopolizes a considerable table area.

In the second arrangement the microscope need not have a mechanical stage, and the objects need not be securely mounted, and very little table room is required. The microscope, moreover, being in a vertical position, it is much more convenient to examine the specimens before the camera is lowered over the eyepiece. There is only one disadvantage to this system, and that is the necessity of standing on a chair or stool to focus the object, but that is a very slight drawback.

Altogether the advantages in favour of the vertical system are overwhelming.

A very simple arrangement for photographing vertically can be made with the assistance of a joiner, at a small cost, not exceeding more than 30/-. Such an apparatus is described in the paper referred to previously.*

A very convenient form of camera and stand is made by Messrs. Nachet & Sons, Paris. An illustration of this camera is

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* *Iron and Steel Journal*, No. 1, 1897.

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shown in Fig. 8, the working of which will be understood with very little explanation.*

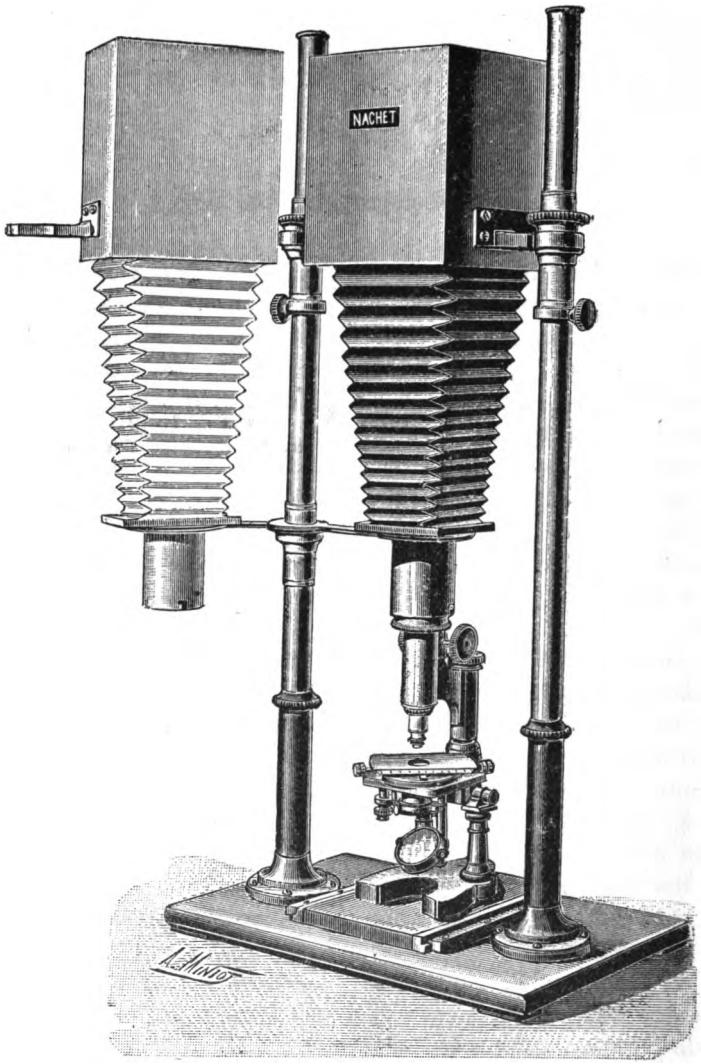

Fig. 8

* A vertical camera of similar construction is manufactured by the Bausch & Lomb Optical Co. of Rochester, N. Y., which possesses some decided advantages over the Nachet type. — Ed.

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It will be noticed that the camera slides up and down on the rigid supports on each side of the vertical microscope. It can also be swung clear of the microscope so that it does not come in contact with the head of the observer when using the instrument during visual examination.

In taking the photograph after having obtained the particular field of the object in position, without removing the eyepiece, the camera is placed in position, the hood lowered over the eyepiece, and the box is raised to the point corresponding to the magnification required. A clear-glass plate is substituted for the ground-glass screen, at the top of the camera. The focussing is effected by applying the eye to a No. 1 eyepiece placed on the center of the glass, and by means of the micrometer screw, adjustment is made till the object is seen most distinctly. The glass screen is removed and a slide with the sensitive dry plate is fixed into place, the slide drawn and the exposure made. The time of exposure depends on the brightness of the object, the quantity of light admitted and on the magnification. In my own practice, using the Empress Isochromatic plates (Ilford), when clear objects are used and a magnification of 100 diameters photographed, from one to five minutes' exposure is necessary to get a good impression, but some objects take more than an hour. The development is done in the usual way, and it is not necessary to describe the process. Personally, I prefer the ready-made solution called "Rodenal," diluted with ten times its own volume of water.

In every camera it is advisable to graduate one of the guides, and mark off the distance from the microscope stage to the back of the camera in inches or centimetres, and to prepare tables with each object-glass correlating the graduation with the diameters magnified. This is done by placing a micrometer slide, 1 mm. divided into 100, on the stage, and allowing transmitted light to pass through it and the microscope, and to fall upon a ground-glass screen in the camera, which has been ruled with lines one centimetre apart. The magnification of the divisions of the micrometer slide can be seen on the ground glass, and the comparison with the ruled lines thereon compared.

By moving the camera box upwards and taking repeated observations, a table can be tabulated for each particular object-glass and eyepiece employed. Allowance, of course, must be

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made for the difference in height of the various thicknesses of the sections on the stage.

APPENDIX.

No. 7.—Lists of Apparatus.

I am frequently asked to advise metallurgical establishments where they can obtain the necessary apparatus required for the microscopic examination of metals, and I cannot go far wrong in giving in this appendix the information so often asked for.

Microscopes.

There are many good microscope makers in England and on the Continent, and it would be unfair to say that one maker is better than another. My advice is: Go to a good well-known maker, tell him exactly what you want, and be guided by him.* It will be necessary, however, to go to the firms referred to in my paper for the special apparatus described. Mr. J. C. Robson, Chemical Dealer, Linthorpe Road, Middlesbrough, and no doubt other chemical dealers in every large town and city, could obtain all the various microscopes and special apparatus required. The following list will give a good idea of what is wanted:

Microscopic Accessories.

1. Three objectives, 1-in., ¼-in. and $^1/_{12}$-in. oil immersion.
2. Three eyepieces, Nos. 1, 2 and 3.
3. Sorby-Beck reflectors, which should be fitted to the 1-in. objective.
4. Beck's reflector for high powers.
5. Nachet's prism reflector.
6. One bull's-eye condenser.
7. Zeiss' object-glass changers.
8. A mechanical stage to the microscope is very useful, but is not absolutely necessary. In a first-class outfit, however, it should be included.
9. A micrometer slide, 1 mm., divided into 1,000.

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* The ordinary microscope with fixed stage is not well adapted to the examination of opaque objects. The special stand with movable stage, now quite universally used in this country, is greatly superior. — Ed.

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Simple Apparatus for Polishing by Hand.

1. A few pieces of hard wood planed smooth; 5 in. by 5 in. by ½ in.
2. Pieces of black unribbed cloth.
3. Do. chamois leather.
4. Do. kidskin.
5. Do. parchment.
6. Do. emery paper (English make).
7. Do. emery paper (French make), used by steel engravers for polishing their plates.
8. A bottle of diamontine powder, used by jewellers for polishing steel; obtainable from T. G. Poole, Jeweller, Middlesbrough, and elsewhere.
9. An ounce of gold rouge.
10. Half-ounce of nitrate of ammonia.
11. A few drawing pins.

Estimated total cost five shillings (5s.).

Good work can be done with this simple set of apparatus.

Mechanical Apparatus.

Messrs. Carling & Sons, Machinists, Middlesbrough, supply the following:

1. Hand hack saw in simple frame* 7s. 6d.
2. Do. do. in rigid frame, with vice for holding metals when cutting out the sections* (spare hack saw blades 5d. each), 16s. 6d.
3. Hand or power hack saw, on stand with vice, manufactured by the Millars Fall Co., Mass., U.S.A.,* £5 5s.
4. Stead's mechanical polishing apparatus, complete with blocks, emery papers, cloth, polishing powders, etc., £3 10s.
5. Carling's mechanical improved polisher, with blocks, £4.
6. Stage temporary mounting arrangement, 2s. 6d.
7. Set of rings in box for mounting the specimens on glass slides, 5s.
8. Plasticene for mounting the objects, 3d. per oz.

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* The above apparatus can also be obtained through Mr. T. Walton, Middlesbrough, or any good ironmonger.

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Cameras.

A simple form of vertical camera can be made at a cost of little more than 25s. A more elaborate stand at any good maker's costs about £5 5s.

Concluding Remarks.

It is easy to spend £50, or even more, on an outfit, but a good, serviceable, complete set of apparatus need not cost more than half that amount. A microscope and accessories costing £15, with the simple polishing apparatus above described, need not cost more than £15 10s.

Microstructure of Steel Quenched from the Melting-Point.— These excellent and interesting photomicrographs were taken by Mr. F. C. Lau.

Fig. 1

Steel containing 2.40 per cent carbon quenched from the melting-point. Magnified 150 diameters.

Fig. 1 illustrates the structure of a bar of steel containing 2.40 per cent of carbon quenched when the end of the bar began

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to melt. 0.092 inch was ground off from the molten end, which was then polished and etched. After polishing, the needles of martensite were well defined, and the section was etched for five seconds in a 10-per-cent solution of nitric acid. The white areas represent cementite. The magnification is 150 diameters.

The characteristics of martensite, first described by Osmond, are here brought out more clearly than we have ever seen them.

By quenching near the melting-point we retain in the cold metal the structural arrangement of martensite on a very large scale, so that a relatively small magnification is sufficient to bring it out. In ordinary quenching (+800° C.), the characteristics of martensite are still retained, but the structural elements are so small that it requires a very high magnification to resolve their arrangement, resulting in a corresponding loss of sharpness and definition; hence, the much greater clearness of Mr. Lau's specimens as compared with photograph of martensite previously published.

It is well to recall here that Prof. Arnold denies any structure to martensite, as Mr. Lau's photograph affords further refutation of such contention.

Fig. 2 shows the structure of the same sample magnified 1,000 diameter. It is one of the best high-power photomicrographs of steel ever published. The markings in the areas of free cementite should be noted. They were not seen under the low magnification of Fig. 1. Mr. Lau thinks they are minute gas cavities.

Fig. 3 exhibits, under a magnification of 250 diameters, the structure of self-hardening steel, quenched from the melting-point. The chemical composition of the steel was as follows:

Carbon	2.29	per	cent.
Tungsten	5.63	"	"
Chromium	0.53	"	"
Manganese	1.87	"	"
Silicon	0.50	"	"
Phosphorus	0.04	"	"

The specimen was etched in tincture of iodine for 25 seconds.

It is seen that the presence of a large percentage of tungsten in a high carbon steel deeply changes its structure; none of the characteristics of martensite are to be found here. The structure

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Fig. 3

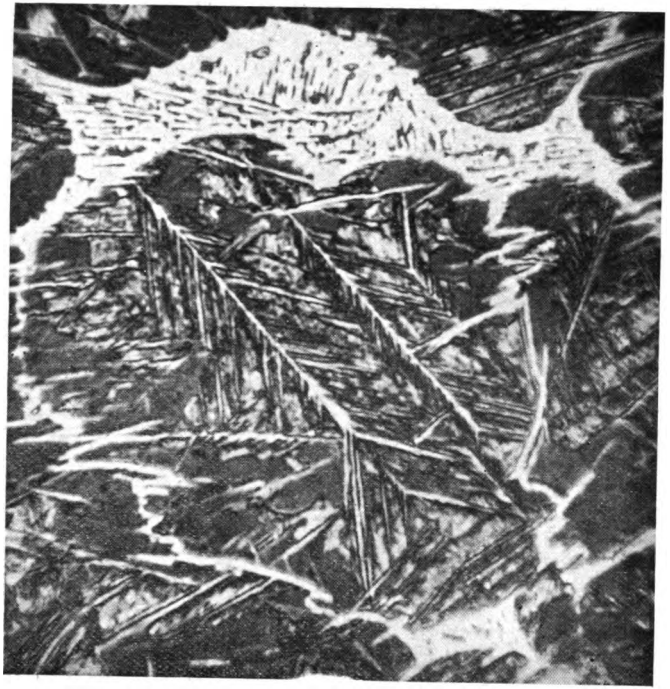

Fig. 2

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is made up if irregular, rounded grains with numerous white areas between them, which probably represent cementite.

The polished section was scratched with a fine point of carborundum, and the marking running diagonally across the photograph shows that the white portion (cementite) was not scratched, while the dark grains are all marked. Even in self-hardening steel, cementite is the hardest constituent.

Soft Steel. — Mr. Sauveur* begins a series of articles on the relation between the structure, treatment and properties of soft steel. In the first installment the microstructure of soft steel and of wrought iron is described and illustrated, a subject with which the readers of this journal are familiar.

The Progress of Metallography in 1899. — Mr. Sauveur† reviews the progress accomplished in metallography during the past year. His review consists of a critical summary of the papers published in *The Metallographist* during 1899.

METALLOGRAPHIC NOTES.

Practical Applications of Metallography. — The study of the molecular constitution of metals, as revealed by etched sections under the microscope, has been considered in many quarters as a laboratory method, of little immediate service in practical work. That this is not the case has been known to a number of conscientious workers in this field on both sides of the Atlantic, and the practical value of the method is well brought out in the papers of Mr. R. Schanzer,‡ recently presented before the Institu-

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* *The Boiler Maker and Sheet Metal Worker.* May, 1900.

† *Mineral Industry,* Vol. VIII, page 702.

‡ Mr. Schanzer's paper will be published in an early number of *The Metallographist.*

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tion of Naval Architects, upon the so-called mysterious fractures of steel shafts.

There has been a number of sudden fractures in shafts and other steel structures which have been subjected to shocks and vibratory stresses, and some of these have been of a most perplexing character, attracting the attention of many of the most authoritative investigators. Most of these fractures represent nothing else than so many cases of brittleness, more or less marked, a phenomenon for which our present knowledge is quite inadequate to account, except in a very limited number of cases.

A very startling feature of such fractures which ought to be noticed is that many sudden breakages in shafts and other structures occur in materials which have been accurately tested before use, and in which the ordinary physical tests uniformly pointed to the best quality of metal. After such a structure has been working continuously for several years in the most satisfactory manner, fracture may suddenly occur, while at the same time no visible evidence of deterioration has appeared up to the very moment of failure, to give warning of the coming danger.

This question of the fatigue of metals has been studied by many eminent authorities, but no definite solution of the matter has yet been reached, and it can by no means be considered as settled. It is, however, possible, by microscopic examination, to discover what changes may have occurred in the physical structure of the metal, and it is the result of such examinations which forms the basis of Mr. Schanzer's paper.

. . . . . .

The serious nature of disasters attending the breakage of shafts, whether at sea or ashore, should make it imperative that every precaution be taken to insure sound metal, and it seems probable that future specifications may include metallographical examinations among the requirements for acceptance. — *The Engineering Magazine,* July 1900, page 588.

The Allotropic Theory of the Hardening of Steel. — Important evidence supporting this theory is offered by Mr. Galy-Acré.* One of the difficulties in the way of the allotropic theory

* Mr. Galy-Acré's article will be published in an early number of *The Metallographist.*

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has been the fact that, in the absence of carbon, sudden cooling which might be expected to retain some of the allotropic high temperature iron has appeared not to cause any hardening. It is true that several investigators have shown that, even if but very little carbon be present, a degree of hardening can be induced altogether disproportionate to the percentage of carbon. Thus I found that a steel containing only 0.022 per cent of carbon had its elastic limit raised 157 per cent by quenching in melting mercury, and that steel containing only 0.009 per cent of carbon had its elastic limit raised 170 per cent by the same means. Unfortunately it was later found that this latter steel contained in addition 0.177 per cent of silicon and 0.22 per cent of manganese, so that the value of this evidence was lessened.

The importance of Mr. Galy-Acré's evidence is in showing that, while iron nearly chemically pure, and wholly free from carbon, can be slightly hardened by sudden cooling, the hardening thus induced passes away with time. He thus shows that the allotropic iron, while retainable even in the absence of carbon, is even in the cold extremely unstable, slipping back to the normal or *Alpha*-state; and he does support that part in the allotropic theory which holds that the action of carbon in hardening is that of a brake, retaining the glass-hard allotropic iron which, but for the carbon, would during even sudden cooling slip back so nearly completely to the normal state that the slight degree of hardening induced by the very small quantity of allotropic iron retained, would escape any but the most careful scrutiny. He makes this explanation more reasonable by showing, first that the allotropic iron is slightly retainable even in the complete absence of carbon; and second, that when thus retained it really is unstable, thus demonstrating that instability which the allotropic theory demands. — H. M. Howe in *Mineral Industry,* Vol. VIII, page 380.

Phosphides and Sulphides under the Microscope. — The paper which was to be presented by Mr. J. E. Stead, at the last meeting of the Iron and Steel Institute, and in which his method of distinguishing the phosphides and sulphides in iron and steel, under the microscope, was to be fully described and illustrated, was at a late date withdrawn by the author and will be read at the Paris meeting of the Institute next September. No time will

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be lost in reproducing Mr. Stead's paper in *The Metallographist.* At the last meeting, however, Mr. Stead said that there were one or two things which he would like to draw attention to, and the first was that by the microscope we could now very easily distinguish the phosphorus compounds in both iron and steel. He had found that iron would absorb about 1½ more per cent of phosphorus in the form of solidified solution. It was exactly like iron in all its properties except that it was exceedingly brittle. The next point of interest was that, if carbon is added to that saturated compound, the carbon throws the phosphorus out of the solution of iron; so that the microscope shows, surrounding the grains of the material, the phosphide of iron that was originally in solution. The method of distinguishing those phosphides was not by any etching method such as had been described repeatedly before these meetings, but by the simple process of placing a polished section upon an iron plate. They all knew how a blacksmith tempered a steel chisel. The phosphides and carbides of iron all behaved differently under identical treatment, and therefore, if they took a piece of pig iron or steel containing phosphorus and polished it so that it looked mirror-like in appearance, they would see no structure whatever in that condition; and, if it was placed upon a hot iron plate and they waited till it assumed a yellowish-brown tint, and then put it to one side to allow it to cool, and then placed it under a microscope, they would find a most marvelous picture. The phosphorus would be found of one tint brilliant in color, the carbide of another and quite different color, and the iron of yet another color. There were the constituents laid side by side, apparently most beautifully painted by nature. Those colors were got by oxidation tints, and it was by those oxidation tints that they could be differentiated.

Metallography at the Technical Schools. — *Massachusetts Institute of Technology.* — Metallography is a subject which has been receiving much attention recently at the Massachusetts Institute of Technology. The science, although established some years ago, has been developed only within the last ten years. The opportunity for the application of metallography in the iron and steel industries is very great, and the Institute of Technology is among the first to introduce a course in the subject for students of chemistry and metallurgy. The lectures on the sub-

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ject are given by Mr. Albert Sauveur. The laboratory work is under the direction of Dr. Henry Fay. Up to the present, most of the laboratory work has been of the nature of investigation by students who have incorporated their results into graduating theses. At present an investigation is in progress the results of which will be of value to makers of boiler tubes. Samples of tubing which have been subjected to a great variety of conditions are being studied. The examination of these specimens will show the effect that the various processes used in the manufacture of such material have on the finished article, and will indicate what treatment is harmful to the life of the tube. Another subject under investigation, which possesses a theoretical interest only, but one of importance, is the isomorphism of selenium and tellurium. The question has been attacked from the standpoint of crystallography, the best method to use in such cases, but with negative results. It is hoped that the application of the principles of metallography will decide the question definitely. Graduates of the Institute who have paid attention to metallography as students are applying their knowledge successfully in the iron and steel industry. They keep in touch with the Institute, and propose practical problems, which have come under their observation, for investigation in the laboratories of the Institute, which are amply equipped for such work. — *The Technology Review,* April, 1900.

The research work of Mr. Stephen Badlam has proved especially interesting and fruitful. It consisted in an experimental study of the relation between the structure, physical properties and heat treatment of very soft steel. We hope to publish his results shortly.

Harvard University. — The Lawrence Scientific School. — Metallography and the physics of metals will be taken up during the next academic year, as a course of research for graduates and other properly qualified students, under the direction of Mr. Sauveur.

The following few lines of investigations, in which fruitful research work might be carried on, are suggested:

I. Relations between the structure of a certain metal or alloy and its physical properties.

II. Influence of the treatment (thermal and mechanical) upon the structure and properties of a certain metal or alloy.

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III. Influence of impurities upon the structure and properties of a certain metal or alloy.

IV. Influence of varying proportions of the constituents of a metallic alloy upon its structure and properties.

V. Influence of the composition and treatment upon the position of the critical points of steel.

VI. Determination of the critical points of special steels which have not so far been determined.

VII. Preparation of a new special steel and investigation of its properties.

VIII. Preparation of a new metallic alloy and investigation of its properties.

IX. Investigation concerning the effect of composition and treatment upon the magnetic properties of steel.

X. Determination of the curve of fusibility of alloys of unknown constitution.

Columbia University. — School of Mines. — Investigations were carried on during the past year by Prof. Howe and advanced students into the metallography and annealing of unforged steel and into the metallography of malleable cast iron.

The valuable thesis work of Mr. R. G. Morse, a recent graduate of the school of mines was published in the last issue of *The Metallographist.*

Metallography at the Watertown Arsenal. — *Watertown, Mass.* — The Annual "Report of Tests" for 1898, recently published, contains twenty-five photomicrographs of steels. They are, however, unaccompanied by any comment, no effort being made to connect their structure with their physical properties, although the metals which they represent had in every case been tested. This is to be regretted all the more that they have at hand exceptionable facilities for a thorough investigation of the relation existing between the microstructure of steel and its physical properties.

The photomicrographs, moreover, indicate, in many instances, imperfect polishing and etching of the metallic sections.

Columbia University Lectures. — Of the five lectures on Metallurgy delivered in March, in New York, at the Museum of

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Natural History, under the auspices of Columbia University, three dealt with metallographic subjects, as follows:

March 3d. — Prof. Henry M. Howe, "The Constitution of Steel as revealed by the Microscope."

March 10th. — Mr. Albert Sauveur, "The Constitution of Metallic Alloys in the Light of Modern Research."

March 17th. — Mr. Henry Souther, "Toledo blades: Rationale of the Procedure in Manufacturing Them and Other Steel. Objects Explained by the Microscope."

Prof. Howe, however, was prevented by illness from delivering his lecture.

Professor Samson Jordan. — In the death of Prof. Jordan which occurred in Paris last February, the World of Scientific Metallurgy loses one of its most distinguished and enthusiastic members. Although not personally engaged in metallographic work, Prof. Jordan followed his development with the greatest interest and with much faith in its industrial value. He was thoroughly posted with every advance made in this field and it is upon his recommendation that the Société d'Encouragement awarded to Mr. Osmond in 1897 the Lavoisier Medal, in recognition of his masterly work dealing with the microstructure and the transformations of iron and steel.

Prof. Jordan was at the time of his death Professor of Metallurgy at the École centrale, at Paris, a position which he occupied brilliantly for many years.

Erratum. — The editor greatly regrets that the following typographical error was left uncorrected in the April 1900 issue of the journal:

The last line of page 98 should be the first line of that page and the last line but one of page 98 should be the last line of page 99.

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FLORIS OSMOND

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The Metallographist.

A QUARTERLY PUBLICATION DEVOTED TO THE STUDY OF METALS, WITH SPECIAL REFERENCE TO THEIR PHYSICS AND MICROSTRUCTURE, THEIR INDUSTRIAL TREATMENT AND APPLICATIONS.

Vol. III. OCTOBER 1900. No. 4.

FLORIS OSMOND.

FLORIS OSMOND was born in Paris, March 10, 1849. He graduated from the École Centrale des Arts et Manufactures, where he studied metallurgy under the late Professor Jordan, whose interest in metallographic researches is well known. After a few months spent in the machine shops of Fives-Lille, Mr. Osmond entered the employ of the Blast Furnaces, Forges and Rolling Mills Co. of Denain and Anzin, which was then adding to its works some Bessemer converters and some open hearth furnaces. Mr. Osmond assisted in starting the steel making department and although devoting his time more particularly to the open hearth furnaces and to the laboratory, he familiarized himself with all the details of a large metallurgical establishment. In 1880, Mr. Osmond joined the metallurgical staff of the famous Creusot works. It is there that he met Mr. Werth, with whose collaboration he wrote his first contributions to the application of the microscope to the study of iron and steel. Drawn towards each other by a similarity of taste, Mr. Osmond and Mr. Werth sought together, along scientific lines, the solution of the daily problems coming to their notice in the actual running of the works, and which so often remained unsolved.

In 1884, Mr. Osmond left the manufacturing line, to devote himself exclusively to the study of scientific metallurgy. It is in his private laboratory, in Paris, that he conducted those admirable researches which have rendered his name famous on two continents.

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In 1888, the French Society for the Encouragement of the National Industry awarded to Mr. Osmond a prize of 3000 francs and in 1895 a second prize of 2000 francs.

In 1897, Mr. Osmond received the Lavoisier medal, in recognition of his masterly presentation and successful defense of the allotropic theory of iron. The Lavoisier medal is awarded every six years by the French Society of Encouragement to the author, whatever his nationality, whose work has most contributed to the progress of French industry. The illustrious Sainte-Claire Deville was the first recipient of the medal. It is an award of which any scientist may justly be proud.

In 1898, The American Institute of Mining Engineers elected Mr. Osmond one of its honorary members.

Mr. Osmond's written contributions to the advancement of metallurgy number over seventy. A complete list of those dealing with metallographic subjects is given below:

The Cellular Structure of Cast Steel. — *Comptes Rendus,* Vol. C, 1885, page 450. (With Mr. J. Werth.)

The Cellular Theory of the Properties of Steel. — *Annales des Mines,* July and August, 1885. (With Mr. J. Werth.)

On the Phenomena which take Place during the Heating and Cooling of Steel. — *Comptes Rendus,* Vol. CIII, 1886, pages 743 and 1135.

Transformations of Iron and Carbon in Iron, Steel, and White Cast Iron. — *Mémorial de l'Artillerie de la Marine,* Vol. XV, 1887, page 573.

On the Microscopical Examination of the Interior of a Gun, etc. — *Mémorial de l'Artillerie de la Marine,* Vol. XVI, 1888, page 89.

Contributions to the Study of Cast Iron. — *Comptes Rendus,* Vol. CVI, 1888, page 1156.

Metallurgical Studies. — *Annales des Mines,* 1888, page 5.

On the New Methods of Hardening. — *Bulletin de l'Industrie Minérale,* 1889, page 1045.

On the Rôle of Certain Foreign Bodies in Iron and Steel. — *Comptes Rendus,* 1890, page 242.

Iron and Steel. — *Lumière Electrique,* Vol. XXXV, 1890.

On the Rôle of Foreign Bodies in Iron and Steel, etc. — *Comptes Rendus,* 1890, page 346.

On the Critical Points of Iron and Steel. — *J. Iron and Steel Institute,* 1890, page 38.

Considerations on Permanent Magnets. — *Philosophical Magazine,* 1890, page 511.

Transformations of Iron and Carbon in Steel. — *Mémorial de l'Artillerie de la Marine,* 1891.

On the Transformations which accompany the Carbonization of Iron by the Diamond. — *Comptes Rendus,* Vol. CXII, 1891, page 578.

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Note on the Microstructure of Steel. — *J. Iron and Steel Institute,* 1891, I, page 100.

Microscopical Metallography. — *Rapports de la Commission des Méthodes d'Essai des Matériaux de Construction,* Paris, Vol. II, page 7.

Microscopical Metallography. — *Transactions of the American Institute of Mining Engineers,* Vol. XXII, page 243.

Alloys of Iron and Nickel. — *Comptes Rendus,* Vol. CXVIII, 1894, page 532.

On the Use of Polishing in the Study of the Structure of Metals. — *Comptes Rendus,* Vol. CXVIII, 1894, page 807.

Contributions to the Study of the Structure of Steel. — *Comptes Rendus,* Vol. CXIX, 1894, page 329.

General Method for the Microscopical Analysis of carbon steel. — *Bulletin Société d'Encouragement,* 1895, page 480.

Application of Microscopical Metallography to the Manufacture of Rails. — *Annales des Mines,* Vol. VIII, 1895, page 153.

Observations concerning Mr. Arnold's paper entitled, "The Influence of Carbon on Iron." *Bulletin Société d'Encouragement,* 1896, page 268.

On the Allotropic Forms of Iron and their Rôle in the Classification of Steels. — *Bulletin Société d'Encouragement,* 1896, page 735.

Note on Mr. Howe's Researches on the Hardening of Steel. — *J. Iron and Steel Institute,* 1896, I, page 180.

On the Structure of Metals, its Origin and Changes. (With Sir W. C. Roberts-Austen.) *Philosophical Transactions,* Vol. CLXXXVII, 1896.

On the Usefulness of Micrographical Analysis in the Metallurgy of Iron. — *Report presented to the International Congress of Applied Chemistry,* 1896.

Contribution to the Discussion of Mr. Sauveur's Paper "On the Microstructure of Steel and the Current Theories of Hardening." — *Transactions American Institute of Mining Engineers,* Vol. XXVII, page 876.

On the Alloys of the Group Silver-Copper. — *Comptes Rendus,* Vol. CXXIV, 1897, pages 1084 and 1234.

Metallography Considered as a Testing Method. — *The Metallographist,* Vol. I, 1898, page 5.

On the Microstructure of Alloys of Iron and Nickel. — *Comptes Rendus,* Vol. CXXVI, 1898, page 1352.

Magnetic Transformation and Hardening of Steel. — *The Metallographist,* Vol. I, page 266.

On the Signification of the Critical Points of Iron and Steel. — *The Metallographist,* Vol. I, 1898, page 271.

The Segregation of Cementite. — *The Metallographist,* Vol. I, 1898, page 356.

The Microscopic Structure of Gold and Gold Alloys. — *Engineering,* Dec. 9, 1898, and Feb. 24, 1899.

The Relation between the Microstructure of Rolled Steel and its Physical Properties. — *The Metallographist,* Vol. II, 1899, page 78.

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The Relation between the Hardening Power and the Retardations of Low-Carbon Steel. — *The Metallographist,* Vol. II, 1899, page 79.

Alloys of Iron and Nickel. — *Comptes Rendus,* Vol. CXXVIII, page 304; *The Metallographist,* Vol. II, page 136.

On the Effect of Low Temperatures upon Certain Steels. — *Comptes Rendus,* Vol. CXXVIII, page 1395; *The Metallographist,* Vol. II, page 261.

On Magnet Steels. — *Comptes Rendus,* Vol. CXXVIII, 1899, page 1513; *The Metallographist,* Vol. III, 1900, page 35.

What is the Inferior Limit of the Critical Point A_3? — *The Metallographist,* Vol. II, 1899, page 169.

Experiments on Alloys of Iron and Nickel. — *Proceedings of the Institution of Civil Engineers,* Vol. CXXXVIII, 1898-1899, page 217.

New Re-agents for the Microscopical Study of Carburized Irons. — *The Metallographist,* Vol. III, 1900, page 88. (With G. Cartaud.)

On the Crystallography of Iron. — *Annales des Mines,* Series 9, Vol. XVII, January 1900, page 110. — *The Metallographist,* Vol. III, 1899, page 181.

Mr. Osmond was the third scientist to take up the microscopical examination of iron and steel, his first article having been written seven years after Professor Martens' first contribution and twenty-one years after Dr. Sorby had called the attention of metallurgists to this method of investigation. As already stated, Mr. Osmond's first two and very important papers were written in collaboration with Mr. Werth. The name of Mr. Osmond is intimately associated with each progress accomplished in metallography and it may be affirmed that no other investigator has contributed as much as he to the advancement of this science.

Mr. Osmond's work bears the stamp of a master mind, thoroughly equipped for original scientific investigation. Erudition, lucidity, logic and brilliancy are some of the qualities which must impress the student of his writings. He possesses in a high degree the fearlessness, thoroughness and caution so necessary for successful research work. Mr. Osmond's well-known modesty adds great charm to his personality and to his writings.

It is not proposed to review here, even briefly, Mr. Osmond's work, but merely to outline its most salient features. His metallographic work may be roughly divided into three groups:

1. His contribution to the study of the microstructure of iron and steel in general, including the technology of the subject and the description and interpretation of the revealed structures.

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2. His discovery of the upper thermal critical points of iron and of low carbon steel and his enunciation and defense of the allotropic theory suggested by these critical points. Under this heading should also be included his study of the changes in certain physical properties occurring at the critical point, especially the changes in the magnetic properties.

3. His study of the microstructure of some special steels — especially of manganese and nickel steels — of their critical points and corresponding changes of physical properties, undertaken with a view of throwing additional light upon the allotropic transformations of iron.

Mr. Osmond's contributions to the technology of the subject have been very important. His paper on "General Method for the Micrographical Analysis of Carbon Steel," for which the Société d'Encouragement awarded him a prize of 2000 francs, will long remain classical. Mr. Osmond was the first to propose the use of tincture of iodine for the etching of polished steel surfaces, and the method is now quite universally used. He also proposed the rubbing of the surface upon a piece of parchment soaked with extract of liquorice roots, but later investigations led him to discard this re-agent, substituting for it a diluted solution of nitrate of ammonium. The polishing in relief, conducted as he described it, yields, with certain classes of metals, beautiful preparations. The fine high-power photomicrographs illustrating some of Mr. Osmond's writings, testify to his skill in all technological details.

Mr. Osmond was the first to demonstrate that the main micro-constituent of hardened steel possessed some characteristic features which entitled it to be considered as a separate entity instead of a mere amorphous mass of varying composition. He proposed for it the name of *martensite* in honor of Professor Martens. To him also we owe the discovery of *austenite,* a constituent found in high carbon steel quenched at a high temperature, of *sorbite,* a form of transition between ferrite and cementite, and of *troostite,* a transition-form between ferrite and martensite.

Mr. Osmond's description of the microstructure of steel and of the relation which it bears to its heat treatment, as contained in several of his papers, should be carefully read by all students of metallography.

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Mr. Osmond was the first, we think, to call attention to the similarity which exists between pearlyte and the eutectic alloys, showing that pearlyte was the true eutectic alloy of steel considered as a solid solution of iron and the carbide Fe_3C, a fact which has proved so fruitful in important deductions.

It is, of course, Mr. Osmond's discovery of two retardations occurring during the heating of iron and of low carbon steel at temperatures higher than that of the previously known critical point of steel (the point of recalescence), and his masterly conception of the allotropic theory based upon this discovery, that stands out as his most important and original work.

We cannot do better in bringing this short sketch to an end than to reproduce here the opinion of one of the foremost American metallurgists, exceptionally well qualified to pass judgment upon Mr. Osmond's work, as it appeared in the *Engineering and Mining Journal* of November 12, 1898.

"The award of the Lavoisier medal to the young but already eminent metallurgical physicist, Mr. F. Osmond, is a matter of no common importance, making, as it does, what we may call the second epoch in the life of the allotropic theory of the hardening of steel, which has justly made him so famous. The fact that the award was made on the warm recommendation of so conscientious and judicious a metallurgist as Professor S. Jordan, gives to it additional weight, and to those unfamiliar with the circumstances vouches for its justice. The first epoch in the life of this theory was its enunciation naturally by Mr. Osmond in 1888. This award may be called the second epoch, because it testifies that, in spite of the bitterness with which this theory has been attacked, its merits and its standing are now officially recognized and stamped by an authority, the weight of which cannot be questioned.

"Mr. Osmond first came into general notice in 1885 through the well-known paper written by him and Mr. Werth, "Théorie Cellulaire des Propriétés de l'Acier," which, even if we disregard the author's youth, must be reckoned a remarkable document. This was followed in 1888 by his pamphlet, "Transformations du Fer et du Carbone dans les Fers, les Aciers et les Fontes Blanches," in which he set forth the allotropic theory. Here was an unknown man who had dared to brush aside the speculations of the eminent metallurgists who had already discussed

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the question, and to assign as the chief underlying cause of hardening a principle which had lain unnoticed beneath the eyes of his really illustrious predecessors. The theory was received with extreme scepticism. But history repeats itself, and Mr. Osmond's theory has outlived ridicule to see its just recognition. It has not only won acquiescence, admiration, and even enthusiastic support; it has established itself as a brilliant conception, based upon a rare grasp of the many and bewildering facts.

"While we do not regard the theory as firmly established, that it is a master-work cannot be questioned, though in its present form its truth is, in our opinion, still open to question. Further and more searching investigation is needed before the theory can be regarded as demonstrated. But we congratulate Mr. Osmond on this well-deserved recognition of his work, which, whether his contentions be right or wrong, must remain a monument of which all metallurgists may well be proud."

The reader's attention is also called to Sir Roberts-Austen's estimate of Mr. Osmond's work, as expressed in his presidential address to the Iron and Steel Institute, published in part in the present number of this journal.

NOTE ON THE STRUCTURE OF PHOSPHORETTIC PIG METALS.*

By J. E. STEAD.

THE general structure of grey and white pig iron has been but slightly studied. There is, therefore, much room for careful research in that direction.

The work of the past has not been of such exhaustive character as to enable correct generalisations to be formed.

What is generally admitted is that in the greyest iron the structure appears to be ferrite cut up by plates of graphite, thin and numerous in close-grained iron and thick and more massive in the more open varieties.

Analysis shows that in such iron there is practically no com-

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* *Proceedings Cleveland Institution of Engineers,* February 26, 1900.

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Fig. 1. Cleveland grey Pig Iron showing graphite plates (black), a ground mass of ferrite and the phosphide eutectic in irregular patches of mottled appearance. Magnified 50 diameters.

Fig. 2. Same as Fig 1, but magnified 250 diameters. The compound character of the eutectic is very evident.

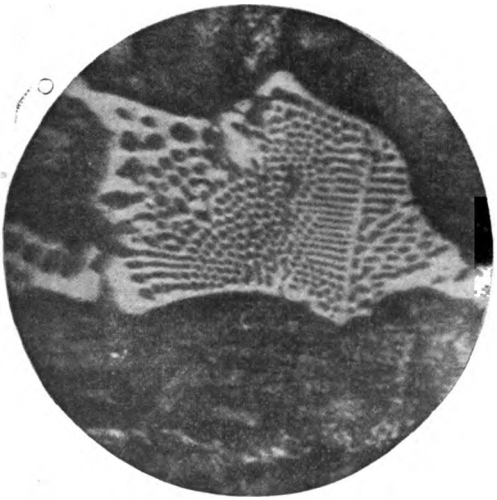

Fig. 3. Highly magnified photograph of one of the phosphide eutectic areas. The ground mass was darkened to more clearly show the eutectic structure. Magnified 400 diameters.

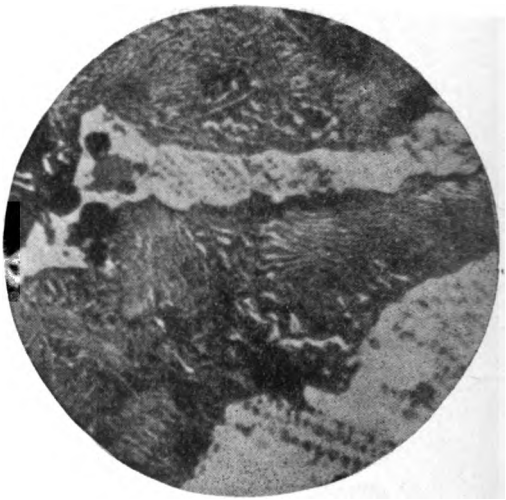

Fig. 4. Cleveland white Pig Iron. The continuous white areas consist of cementite, the mottled areas of pearlyte and phosphide eutectic. The section was etched with iodine, which imparts a similar appearance to both constituents.

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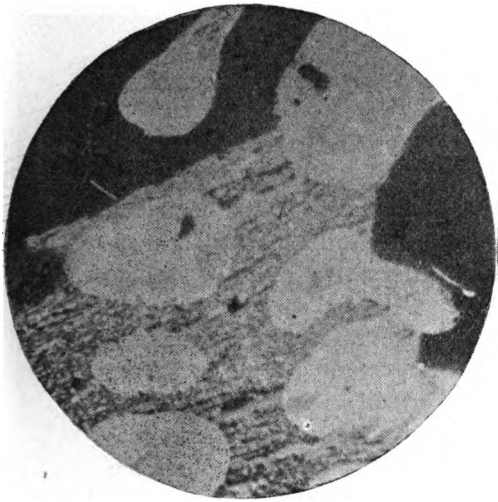

Fig. 5. Basic Pig Iron, containing 2.90 per cent of phosphorus. Structure developed by "heat tinting" — the dark flat areas are cementite.
The globular areas are pearlyte — the mottled areas are the phosphide eutectic.
Magnified 200 diameters.

Fig. 6. Iron, containing 6 per cent of phosphorus and 1.60 per cent of carbon, etched with iodine — the white bands are cementite, the mottled ground mass consists of the phosphide eutectic, the dark areas are pearlyte.
Magnified 100 diameters.

Fig. 7. White Hematite Pig Iron, containing 0.04 per cent of phosphorus, etched with iodine — the dark areas are pearlyte, the white areas cementite.
No phosphide eutectic can be detected.
Magnified 100 diameters.

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bined carbon, therefore practically no pearlyte and no cementite is present.

As the combined carbon increases, at first pearlyte appears, and afterwards, when the quantity is considerable, massive cementite becomes evident in which the carbon exists in the combined state. In white iron the structure appears to be essentially massive cementite (Fe_3C) and pearlyte (alternative layers of carbide of iron and ferrite).

These statements, I think, will be admitted by all metallographers who have made an elementary study of the structure of such irons as are made from Cumberland or Spanish iron ores.

During the last twelve months I have made special examinations to ascertain if there was any material difference between the structure of phosphorettic and non-phosphorettic pig irons.

The results obtained have proved that the difference is considerable.

The phosphorus in phosphorettic pig irons exists as phosphide of iron in a eutectic as a definite chemical compound having the formula Fe_3P, and this compound exists in isolated segregated masses, easily identified in the grey irons but less so in white irons.

Practical men know very well that it is impossible, from the appearance of the fracture of pig metal, to tell whether a cast iron is phosphorettic or not.

By polishing a chip of the iron and etching it with iodine solution, or by heat tinting, it is now quite easy to see under the microscope the phosphide areas and to make an approximate estimate of the quantity of phosphorus present.

A series of specimens of grey iron containing 1.5 per cent, 1.00 per cent, 0.50 per cent, 0.25 per cent, 0.10 per cent, and 0.05 per cent phosphorus were prepared. After polishing they were mounted. The slides were not marked, so that it was impossible to tell which was which; yet it was easy to arrange them in sequential order on examination under the microscope.

It was also possible to detect isolated specks of phosphide in slowly cooled grey metal containing less than 0.05 per cent phosphorus.

The portions last to solidify were those which contained the most phosphorus, and, as one would expect, the phosphide areas were always found in those positions, where, when solidifying,

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the first portions to freeze forced them. They were generally found farthest removed from the plates of graphite in grey iron, the graphite representing the portions first to fall out of solution.

The phosphide in white irons occupied areas between the massive plates of cementite, which latter first crystallised, leaving the fusible phosphorus eutectic compound to solidify in those areas whither it was driven by the first constituents which fell out of solution.

In any of the ordinary Cleveland white pig irons it is easy to identify the massive carbide plates, the pearly constituent of Dr. Sorby (perlite, pearlite, pearlyte), the second pearly constituent or eutectic of phosphorus, and also the sulphides.

The best way of treating a specimen to enable one to identify these constituents is to heat the polished metal upon a hot plate till it assumes a purple tint to the naked eye. The various constituents oxidise at different rates, and assume corresponding colored films. Heated in this way pearlyte assumes a blue tint with brown red worm-like layers of carbide of iron.

The massive carbide takes a fine red brown tint, the phosphide of iron a pale yellow color.

The sulphides do not apparently change color, but are easily detected, as they appear of a slate color and exist in isolated segregations, very often in idiomorphic crystals.

These colors only appear in this order if the heating has been correctly done. By longer heating the colors advance in sequentially progressive steps forward through yellow, brown, orange, red, purple, blue, to slate grey.

Such specimens properly heat tinted are magnificent microscopic objects.

The ordinary methods of etching do not enable one to find any difference between carbides and phosphides, and in the past some metallographers, myself included, have mistaken the one for the other.

The heat tinting process enabled them to detect with certainty the phosphides.

The phosphorus eutectics of white iron are quite different from those of grey pig, both in form and composition.

Variations in the proportion of silicon, manganese, etc., and combined carbon appear to affect the character of the phosphorus eutectics.

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These various fusible compounds are being carefully studied.

In a paper on "Phosphorus in Cleveland Ironstone and Iron," 1875-76, experiments are described in which Cleveland cast iron had been subjected to hydraulic pressure after the greater mass had become solid. The last portion to solidify was by that means squeezed out of the pasty mass. The results are here reproduced as they bear directly on the remarks just made.

The analysis of the original iron together with that of the fusible compound is as follows: —

	Original Metal.	Portion squeezed out.
Iron (by difference) . .	92.36 per cent	89.70 per cent
Carbon	2.84 „	2.00 „
Manganese	0.28 „	0.24 „
Silicon	3.01 „	1.51 „
Sulphur	0.05 „	0.03 „
Phosphorus	1.46 „	6.52 „
	100.00 „	100.00 „

The metal squeezed out may be truly regarded as the eutectic of Cleveland pig iron.

In concluding this note it may be considered as established that in pig irons the phosphide, if not entirely, is at least practically, in a separate state in segregated masses in eutectics in the state of Fe_3P, a definite chemical compound of iron and phosphorus.

A more comprehensive research on "On Iron and Phosphorus" is in course of being completed, the results of which will be presented to the Iron and Steel Institute.*

* This paper has now been published and will appear in an early number of *The Metallographist.* — Ed.

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THE INTERNAL ARCHITECTURE OF STEEL.*

By J. O. ARNOLD.

THE theory of the close analogy of steel to a crystallized igneous rock was enunciated by Dr. Sorby in 1864, and has by the labors of many distinguished metallurgists been fully established. Amongst those who have contributed to this result may be specially mentioned the names of Behrens in Holland, Martens in Germany, Sauveur in America, and Stead in England. A type illustrating this analogy is granite. In granite we have segregated specific constituents of approximately definite composition and hardness, viz., mica, felspar, and quartz. So in very hard steel we have of fairly constant composition and hardness the constituents pearlyte and cementite, or in softer steels the constituents pearlyte and ferrite. All these constituents were discovered by Sorby and named by Howe. The case of steel, however, is far more complicated than that of granite, so that the analogy only holds good up to a certain point. To exhaustively treat the internal architecture of steel in a single paper is impossible, therefore I purpose to present only a skeleton outline of the subject. In the infancy of micrographic analysis some metallurgists vastly underrated its usefulness. At the present time there is a tendency on the part of certain workers to overrate its capabilities and to come to specific conclusions concerning the nature of steel on micrographic data alone. Both these extreme views are erroneous. Micrographic analysis of steel is of great importance, and is likely to become still more important in the future, but it must be remembered that *per se* it is not only of little value but possibly misleading. Micrographic analysis should be strictly an auxiliary to aid in coming to a correct idea as to the nature of a steel which has failed and caused a disaster, and its indications are only valuable when correlated with the analysis and mechanical properties of such steel. The elements (other than iron) of ordinary steels, the compounds of which can be directly recognized by the microscope are only two in number, viz.: carbon and sulphur. Of these very small percentages can be readily detected as carbide or sulphide. The latter constituent,

* Read in the Mechanical Science Section of the British Association for the Advancement of Science.

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however, has been curiously neglected by most metallographists. The elements phosphorus, silicon, chromium, nickel, tungsten, etc., can only be recognized indirectly by their influence on the carbide of iron and the crystallization of the mass. It is also necessary here to recall attention to the fact that at a yellow heat, 1100° C., the elements of steel have been divided into migratory or mobile and fixed or immobile elements. The mobile elements are carbon, sulphur, phosphorus, and nickel. It is not yet definitely known if fixed elements such as manganese and silicon may, in combination with carbide or sulphide of iron, become migratory. The micrographic features marking the passage of pure iron into steel as the carbon rises are shown by the series of drawings exhibited. Pure iron appears as polygons formed by interfering or allotrimorphic crystals belonging to the cubic system (Fig. 1). From a mechanical point of view the internal structure of these polygons (which are sometimes incorrectly termed "grains" or even "blobs") must be considered to be built up from crystalline and not from molecular units. On the addition of carbon to the iron to the extent of, say, 0.3 per cent, one-third of the iron is converted into "true steel" (Fig. 2). In unhardened steels this "true steel" or pearlyte consists of 87 per cent of iron holding in suspension 13 per cent of carbide of iron Fe_3C in the forms of minute plates or granules. These areas are on etching with very dilute nitric acid automatically stained dark brown with a carbonaceous coloring matter liberated by the acid, the iron or ferrite remaining bright. On adding 0.6 per cent (Fig. 3) of carbon the structure contains two-thirds of "true steel"; in other words, whilst the 0.3 per cent steel consisted of one-third pearlyte in a matrix of two-thirds ferrite, the 0.6 per cent steel consists of one-third ferrite and two-thirds pearlyte. On adding 0.9 per cent carbon (Fig. 4) the iron becomes "saturated," and the whole structure is pearlyte, or "true steel"; on adding, say, 1.2 per cent carbon (Fig. 5) the metal is found to be super-saturated, and consists of a matrix of pearlyte forming large cells, the walls of which consist of cementite, that is, the 0.3 per cent of surplus carbon combined with its equivalent of iron to form the compound, normal carbide of iron, Fe_3C. Saturated and super-saturated steels are used mainly for machine tools, whilst sub-saturated steels are employed for structural purposes, and are hence of vital interest to the engineer. Structural

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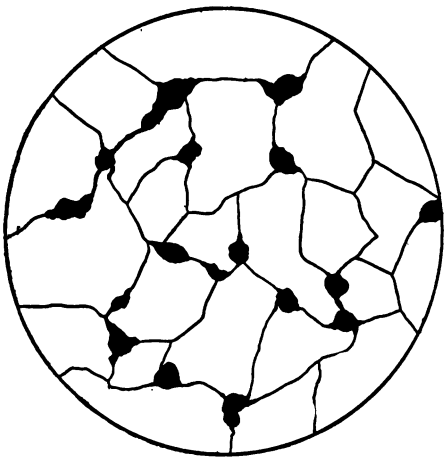

Fig. 1.
Carbon 0.08 per cent.

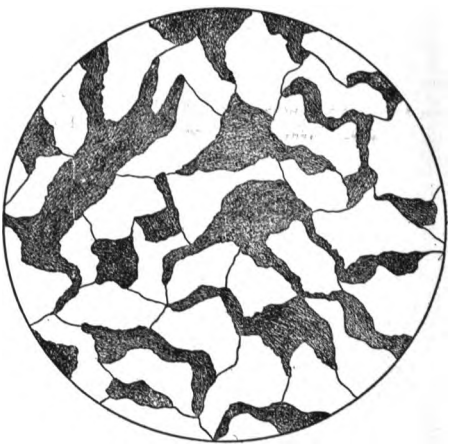

Fig. 2.
Carbon 0.38 per cent.

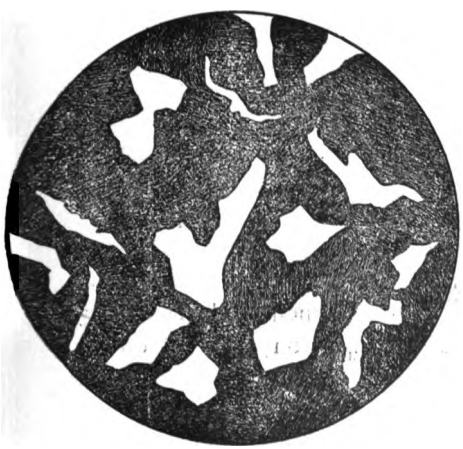

Fig. 3.
Carbon 0.59 per cent.

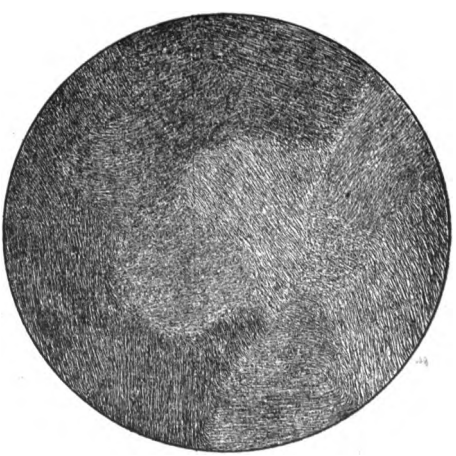

Fig. 4.
Carbon 0.89 per cent.

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steels are usually low in silicon, sulphur, and phosphorus, but their properties are not identical with pure iron and carbon steel from the fact that they contain about 0.5 per cent of manganese. To understand the influence of the manganese it is, however, necessary to first of all examine the properties of unsaturated steels consisting of nearly pure iron and carbon. Nearly pure iron (Fe 99.8 per cent), when fused and afterwards forged, has under normal conditions a maximum stress of about 20 tons per square inch, an elongation on 2 inches of about 50 per cent, and a reduction of area at the point of rupture of about 80 per cent. Its

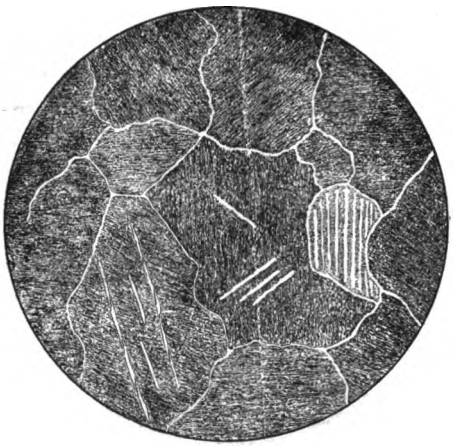

Fig. 5.
Carbon 1.20 per cent.

Fig. 6.
Same Steel as that of Fig. 2, annealed.

elastic limit is low, being only about 11 tons per square inch. The addition of 0.3 per cent of carbon (a typical mean percentage for structural steels) greatly modifies the foregoing figures. The elastic limit rises to about 17 tons per square inch. The stress to about 27 tons per square inch. The elongation per cent on 2 inches falls to about 37 per cent, and the reduction of area to about 60 per cent. If, however, a normal steel giving the test just specified be subject to a drastic annealing, its mechanical properties (without alteration of chemical constitution) are profoundly modified. The limit falls to 9 tons, the maximum stress to 23 tons, whilst the ductility as measured by elongation and

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contraction of area remains practically unchanged. The chemical composition being the same, it remains for the microscope to determine the cause of the decisive fall in the limit and ultimate stress caused by annealing. A glance at Fig. 6 will show how remarkable is the change in the internal arrangement of the constituents. The long, straggling veins, or small patches of granular pearlyte have changed to compact laminated areas partially surrounded by cell walls of cementite. This structure reduces the extra tenacity conferred on the iron by the carbide to its minimum. In fact, it may be taken as a law that the more finely divided and diffused the carbide, the higher the limit and maximum stress. The case of a steel with 0.3 per cent of carbon, containing also about 0.6 per cent of manganese, will next be considered, such material being in regular use practically. The influence of the 0.6 per cent of manganese is very remarkable. It raises the elastic limit from 17 to 25 tons, the ultimate stress from 27 to 35 tons, whilst the elongation on 2 inches falls from 37 to 29 per cent, and the reduction of area from 60 to 47 per cent. The influence of annealing on the manganiferous metal is also very marked, but differs in its nature from the effect produced on pure iron and carbon steel; because, whilst a decisive fall in limit and stress takes place, this (as is not the case in the carbon steel) is accompanied by a distinct increase in ductility. The limit and stress fall about 7 tons each, the elongation rises 6 or 7 per cent, and the reduction of area about 10 per cent. These mechanical differences are co-related with distinct alterations of microstructure. The differences between the carbon-manganese steel and the nearly pure carbon steel are due to two causes — *a.* The stiffening of the iron matrix by the manganese diffused through it; *b.* the fact that the mobility of carbide of iron is much retarded if the iron matrix contains an appreciable quantity of manganese (say, 0.5 to 1.0 per cent). The fact specified under *b.* causes the carbonized regions of the manganese steel to be distinctly larger in sectional area than in the pure carbon steel, and also keeps the carbide in a finer state of division, practically preventing any formation of carbide laminæ. On annealing, however, the factor *b.* is almost eliminated, and the difference in the mechanical properties of the respective annealed steels are mainly due to the influence of the manganese on the iron matrix; that is to say, the limit and stress are greatly raised whilst the ductility is relatively

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little reduced. It should be carefully borne in mind that the foregoing data have reference only to worked steels, and not to steel castings which present quite different properties, and generally are not comparable with forged steels.) Having briefly sketched the chemical, mechanical, and micrographic features of typical constructional steel, the author has to call attention to matters of vital importance in connection with such steels, and also to confidently state that the mechanical tests specified by engineers do not invariably form a guarantee that such a steel is fit for its purpose, or that it will not fail in work, causing possibly a deplorable loss of life. Both engineers and metallurgists have now to face the uncomfortable fact that a steel may bend double cold without any sign of rupture; it may stand a satisfactory tensile test showing a good elastic limit, a sufficiently high ultimate stress and at the same time possess a high ductility as measured by elongation and reduction of area per cent; then nevertheless such steel (or indeed wrought iron) may suddenly rupture without elongation under a stress far below its elastic limit. The explanation of the foregoing facts is that mechanical tests cannot determine whether or no the structural constituents of a steel will cleave under long continued and severe vibratory stresses. It is to the microscope that engineers must look for this information. Rupture under vibration may be of two classes: 1. Interconstitutional cleavage taking place along the junction lines of two constituents; 2. inter-crystalline cleavage taking place along the junction lines of like crystals. In addition to the foregoing, Stead has shown in an isolated case that the brittleness of some steel sheets was due to an internal cleavage of the secondary crystals of iron, resembling a true mineral cleavage. In this case, however, the mechanical tests detected the brittleness of the material previous to Stead's able micrographic demonstration of its cause, and such cases are rare and unconnected with the phenomenon at present under consideration. The metallographists' knowledge of the structures liable to sudden fracture without elongation under vibratory stresses is by no means complete, but the following appearances are, in the author's experience, indicative of dangerous steels: — *a.* "Loose" intercrystalline ferrite joints, i.e., junctions which develop rapidly with very slight etching; *b.* a "trellis work section," in which the laths are ferrite or iron, and the spaces are pearlyte or steel; *c.* cellular structures of

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any kind. With reference to type *a.* it is not yet clear if such joints are "loose" owing to actual intercrystalline spaces, or are due to the mode of intersection of the axes of the secondary crystals (building up the large allotrimorphic crystals), which secondary crystals have different orientations in adjacent primary crystals. In either case the cause of the phenomenon is thermal, and is probably often due to too high an initial temperature. Type *b.* is obviously liable to inter-constitutional cleavage between the long lines formed by the junctions between the iron and steel. Type *c.* is extremely dangerous not only from the palpably weak arrangement of the constituents, but also from the fact that frequently the co-efficient of contraction of the cells is not identical with that of the cell walls. A well-known example of the fragility of such structures is, say, a No. 5 cemented bar which has pearlyte cells bound together by thick cementite walls; the latter contract during cooling to such an extent that the mass is very feebly held together, and blows from a hand hammer will break up a three-inch by one-half-inch bar with ease. The constituent sulphide of iron seems only to exist in steels very low in manganese. It is readily recognized by its pale brown color, which sharply contrasts with the brilliant white of carbide of iron. The sulphide may exist in three distinct forms: 1. As globules or ovoids; 2. as thin laminæ of an irregular oval shape; 3. as cell walls presenting in section a mesh-like appearance. The first named form is that in which sulphur exerts the least injurious effect. The second form is produced when an ingot is reheated to such a high initial temperature that the globules of sulphide become liquid and are hence rolled out into laminæ just as the slag globules in wrought iron are. This form must be regarded as dangerous, more so than the corresponding slag laminæ in wrought iron. The last named are fixed, whereas the mobility of iron sulphide at a full red heat may cause it to invade and render rotten the iron in its vicinity.

The third or meshed form rarely occurs in forged steel, though frequently in small steel castings before annealing. It is invariably a most dangerous constituent. To take an extreme experimental example, 1 per cent of sulphur arranged in this manner entirely destroys the ductility of iron and reduces the ultimate stress to the absurd figure of two tons per square inch. The micrographic recognition of sulphur also presents other

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peculiar features. Before etching, if the plane of section be above the maximum diameter of the globule, the constituent appears as a pale brown structureless plate. If, however, the plane of section be below the maximum diameter, the piece drops out during the polishing and the cavity fills with rouge. On etching all but very compact masses of sulphur are dissolved out, leaving a dark brown residue. During the polishing, particles of sulphide remaining *in situ* become slightly in relief owing to their hardness. This raised point protects a circular region of iron in its vicinity from the action of the polishing block, and hence such iron remains bright and metallic. On etching, the very dilute nitric acid employed liberates H_2S from the sulphide, and the gas produces iridescent color films on the bright iron. The place formerly occupied by the sulphide now appears as a dark spot in the middle of the colored circle or streak. On slightly repolishing the section on the rouge block, all sulphide cavities are filled with rouge and appear as bright red spots, streaks, or meshes, distinctly locating the sulphide, but considerably exaggerating its sectional dimensions. In steel containing manganese, sulphur appears as dove-colored MnS, nearly always in globules or ovals, sometimes as oval laminæ, but in the author's experience very rarely as meshes, manganese seeming to almost entirely prevent the formation of cell walls of sulphide.

In concluding the scanty and suggestive outline of the subject sketched in this paper, the author wishes most strongly to deplore the manner in which pure physicists continue to ignore the work of steel metallurgists on the physics of iron and steel, the result being that what might have been very valuable researches on the magnetic properties of steel cannot be accepted as reliable by the metallurgist, because he knows that although the researches have been conducted with much skill, nevertheless at the outset the physicist almost invariably neglects the most elementary metallurgical precautions simply because, as a doctor of science remarked to the author, "physicists cannot find time to read the technical papers." The obvious retort to such a plea is, that physicists can often find time to make elaborate researches under conditions which render even approximate accuracy impossible. To put the foregoing indictment into definite and concrete form: take a series of steel bars, upon which determinations of permeability and permanent magnetism are about to be made for cor-

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relation with carbon percentages. Many such determinations have been made, but the results are of more than dubious accuracy. The analyses are given, but it is seldom stated whether the drillings for analysis were taken from the bar or the ingot. The previous thermal history of the steel is never stated, and, indeed, is generally unknown. Yet it is of the greatest importance that such data should be given. The analysis of the ingot and bar in high carbon steels are never identical. Then again, no micro-sections of the bars or rings ever seem to be made, and this is a feature of vital importance to secure reliable results. The average bar of hard steel as it comes from the rolls consists of an attenuated outer ring of nearly carbonless iron, then a ring of soft steel, then a ring of saturated steel, then the true supersaturated structure, say of 1.5 steel. As often as not, the physicist gets the bars rolled nearly to size, cleans them up and reports the results obtained from such a concentric compound bar as those correlated with a steel throughout which is evenly diffused, say 1.5 per cent. It is not necessary to further comment upon the hopeless initial error involved in such proceedings. How to remedy such a deplorable state of affairs is not clear. Pure scientists will not read the work of the applied scientist because it is published in technical papers. Purely scientific societies, such as the Royal and Chemical, will not recognize applied work nor will they allow papers on such subjects to be published in their proceedings. The net result is that the pure physicist and the applied physicist, instead of working together, the one supplementing the work of the other, are at cross purposes, and such a state of things cannot be conducive to the advancement of science.

ON THE CRYSTALLOGRAPHY OF IRON.

By F. OSMOND.

(*Concluded from page 219.*)

Crystallography of Beta-Iron.

THE opportunities to study the crystallography of *Beta*-iron are fewer than in the case of *Gamma*-iron. *Beta*-iron cannot be retained *integrally* at the ordinary temperature. Its zone of stability is located, in pure iron, between 750° and 860° C.,

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or thereabout, and this zone becomes the more narrow the nearer the carbon content approaches 0.80 or 0.90 per cent, being finally reduced to a point in the case of more highly carburetted iron. When carbon is not present, however, those substances whose atomic volume is greater than that of iron, especially silicon, phosphorus, and aluminum, when present in sufficient proportions, eliminate the point A_3, or, in other words, extend the zone of stability of *Beta*-iron from 750° to the melting-point.

The methods which may be used to determine the crystalline forms of *Beta*-iron are, therefore, the following:

1. Gather the crystals which might exist in the cavities of alloys of iron deprived of the point A_3; this, however, has never, to my knowledge, been done.

2. Etch the same alloys above 750°, or pure iron between 750° and 860°, which also remains to be done.

3. Examine the structure of steels deprived of the point A_3, because it is supposed that the crystalline forms assumed at a high temperature when the iron was in the *Beta*-state would remain discernible, although *pseudo-morphic,* at the ordinary temperature.

This last method has been applied with much skill and success by Mr. Stead.*

It is the alloy containing 4 per cent of silicon which gave the best results, after having been etched with dilute nitric acid (10 parts of acid to 90 of water, for instance) during a sufficiently long time (10 minutes to 3 hours).

This alloy is made up of small cubes piled up and whose orientation remains the same in the same grain, but varies from one grain to another. Fig. 48 shows, under a magnification of 130 diameters, a section of a grain nearly parallel to one side of the cubes. The alloy contained 4 per cent of silicon and 0.02 per cent of carbon. Fig. 49 exhibits, under the same magnification, the appearance of a grain cut along a diagonal of the cubes. Mr. Stead succeeded in detaching, by cleavage, perfect cubes in a sample of phosphorettic iron very slowly cooled (see Fig. 50). The iron contained traces of carbon and 0.75 per cent of phosphorus.

I infer from the above that *Beta*-iron crystallizes in the cubic system, because:

* *Journal Iron and Steel Institute,* 1898, I, page 145, and 1898, II, page 137. — *The Metallographist,* Vol. I, page 289, and Vol. II, page 85.

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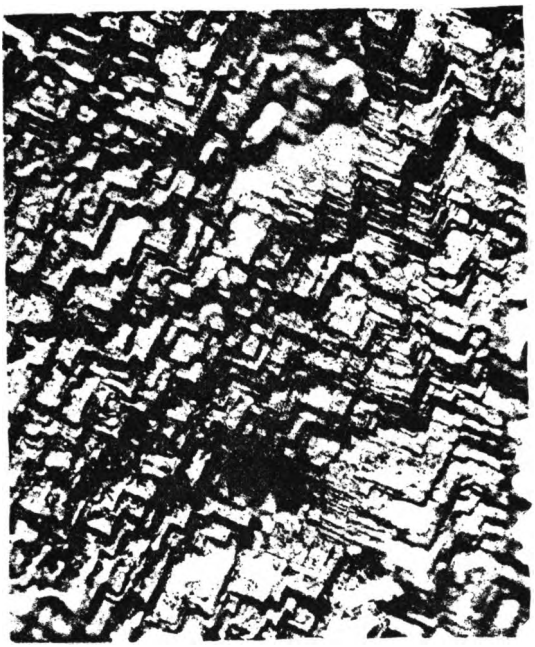

Fig. 48

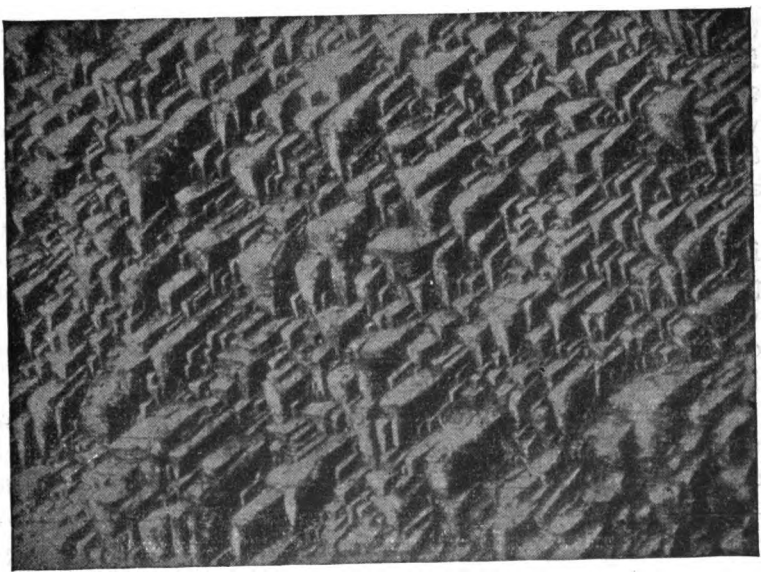

Fig. 49.

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1. The relatively large size of the crystals leads us to infer that they were formed at a high temperature, and in the samples examined, at least in the silicon alloys, it is the form *Beta* which is stable at a high temperature, up to the melting-point.

2. These alloys of silicon and phosphorus, which have no A_3 point, do not change their structure by reheating, according to Mr. Stead's observations; from which it appears that the crystalline forms found at the ordinary temperature are, indeed, those that belong to the solidification period, that is, to the period of stability of *Beta*-iron.

As the actual molecular condition, however, at the temperature at which the etching was conducted, is in reality the *Alpha* condition, these conclusions may not be conclusive.

Crystallography of Alpha-Iron.

It would seem, at first sight, as if the crystallography of *Alpha*-iron must be the easiest to study, seeing that iron as we handle it is, in general, *Alpha*-iron.

If we consider, however, that *Alpha*-iron does not appear during cooling before a temperature of about 700° C. is reached; that the molecular mobility must be feeble at a temperature so far removed from the melting-point, and that we are dealing with a solid substance, it must be admitted that the conditions are not very favorable to crystallization, and it may be feared that the true forms of *Alpha*-iron will be hidden by previous forms acquired under other states.

As a matter of fact, if a polished surface of practically pure iron be properly etched, cubic markings are detected, either depressed or in relief, in those grains which are properly oriented with regard to the plane of the section, and these markings, except in size, are altogether similar to those found by Mr. Stead in the case of ferro-silicon alloys.

Mr. Andrews,* if I am not mistaken, was the first one to reveal such structure by etching with nitric acid some sections cut from large masses of forged iron. Since then it has been

* *Proceedings Royal Society,* Vol. LVIII, page 59.—*Engineering,* Vol. LX, page 88.—*Journal Iron and Steel Institute,* 1895, II, page 542, and 1898, II.

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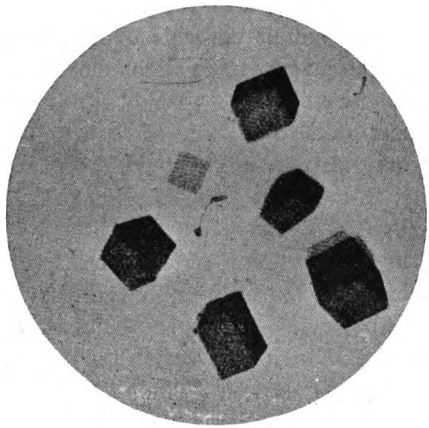

Fig. 50.

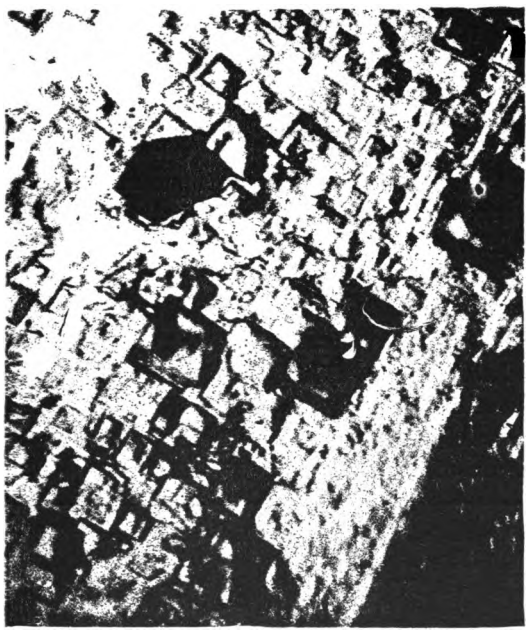

Fig. 51.

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detected and described by Mr. Martens,* by Mr. Stead,† bv Mr. Saniter,‡ and by Mr. Heyn,§ who has obtained very good results by using a solution of double chloride of copper and ammonium (1 part of salt to 12 to 30 parts of water; 1 to 25 minutes' immersion). A prolonged etching (15 or 25 minutes) in dilute sulphuric acid (1 part acid to 4 parts of water) at a temperature of about 50° C. may also be used, in which case the preparation should be washed in a 20 per cent solution of nitric acid for 20 or 30 seconds.

Fig. 51 is the reproduction of a photomicrograph taken by Mr. Heyn and which has not been published elsewhere. It shows the appearance of the ferrite of a soft steel containing about 0.25 per cent of carbon and in a cast condition. It was etched five minutes with the double chloride. The photograph, which, owing to its magnification (1650 diameters), is remarkable, gives a very good idea of the cubic markings of ferrite.

Although Professor Martens and Mr. Heyn hesitate to express an opinion with regard to the meaning of these appearances, and although Mr. Heyn insists upon the difference between the etched figures and the true crystals, I believe, with Mr. Andrews and Mr. Stead, that they actually represent negative or positive crystals.

These crystals are, in my opinion, due to *Alpha*-iron, because I always find them in ferrite, even after forging to a relatively low temperature, which treatment must certainly interfere with the crystallization in the *Gamma* and *Beta* states; and because they are also found in ferrite when the metal contains a relatively large amount of carbon, in which case the zone of stability of *Beta*-iron is very narrow.

The second argument, however, is weak, and it is probably somewhat arbitrary to attribute the same cubic forms now to *Beta*-iron in the silicon alloys of Mr. Stead, now to *Alpha*-iron in ordinary ferrite, although their respective volumes are roughly in the proportion of 1000 to 1.

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* *Mittheilungen aus den k. k. technischen Versuchsanstalten,* Vol. XIV, page 89.

† *Journal Iron and Steel Institute,* 1898, I and II.

‡ *Journal Iron and Steel Institute,* 1898, I.

§ *Mittheilungen aus den k. k. technischen Versuchsanstalten,* 1898, page 310.

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Indirect Proofs of the Isomorphism of Beta-Iron and Alpha-Iron.

If the slender thread of my reasoning is nowhere broken, both *Beta*-iron and *Alpha*-iron crystallize in perfect cubes and are strictly isomorphous; the transformation A_2 is not accompanied by any change of the planes of symmetry.

It is no longer necessary to take into consideration Professor Arnold's experiment, by which the elongated grains of ferrite (due to forging at a low red heat) were made to assume again their primitive forms through reheating to 750° C. This experiment seemed to indicate a crystalline change occurring at the point A_2, which, on that account, Professor Arnold called the *crystallization point of iron.** Mr. Stead has shown more recently that, in order to cause the grains of ferrite distorted by cold work to resume their equiaxial forms, it is enough to heat the metal, during a sufficiently long time, to a temperature which need not exceed 600° C., when the whole, or nearly the whole, of the iron is in the *Alpha* state.†

The purely *crystallographic* facts, however, just given in support of the isomorphism of *Beta*-iron and *Alpha*-iron are not very conclusive. I shall therefore use another argument, altogether independent, which renders this isomorphism probable.

Mr. H. Le Chatelier, in studying, in his *Recherches sur la Dissolution,*‡ the laws of allotropic transformations, distinguishes the following transformations:

1. The allotropic transformations in the crystallized condition which take place abruptly and in a reversible manner, and which correspond to a series of pressures and temperatures related to each other by the equation of Clapeyron-Carnot.

2. The allotropic transformations in the amorphous condition which take place progressively and continuously in a limited range of temperature.

"This way of looking at the phenomena of allotropic transformations," adds Mr. Le Chatelier, "is perfectly justified by the general laws of chemical mechanics. Any reversible, chemical transformations, produced by a variation of temperature, are

* *Journal Iron and Steel Institute,* 1894, I, page 132.

† *Loc. cit.*

‡ *Annales des Mines,* February, 1897.

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sudden when the final products of the reaction remain in the condition of a mixture, being merely juxtaposited, and such a condition is fulfilled in allotropic transformations in the crystallized state; the chemical reaction is, on the contrary, progressive when the final results of the reactions are intimately mixed, and this condition is fulfilled in allotropic transformations in the amorphous state."

If these considerations were to be taken in a narrow sense, it would be difficult to apply them to the transformation of *Beta*-iron into *Alpha*-iron; for, if *Beta*-iron and *Alpha*-iron are crystallized, their transformation should be sudden, which is contrary to our observations; and if their transformation is progressive, they should be amorphous, which is also contrary to our observations.

It is, however, easy to escape this dilemma if the above propositions are worded somewhat differently, and this wording Mr. Le Chatelier, as far as I can judge, has adopted in his most recent communications.

It is sufficient to note that the reversible changes of state, giving to the expression " change of state " its broadest meaning, take place at a constant temperature only when the pressure remains constant, and that the temperature at which they occur is variable if the pressure varies.

We are led to ask, therefore, what are, in the case of allotropic transformations in the solid state, the conditions which are necessary for the pressure to remain constant.

We must, at the outset, consider, besides the atmospheric pressure, which remains practically constant, two kinds of pressure which are liable to vary: the osmotic pressure and the mechanical pressure.

1. The osmotic pressure can only remain constant if it does not exist, that is, if the final products of the transformations are insoluble in each other or, in the case of crystallized bodies, do not form any isomorphous mixtures. Were it not so, the osmotic pressure would vary at every instance of the concentration.

2. The mechanical pressure results from the change of volume which accompanies (or may accompany) the transformation. This pressure also, in order to be constant, must be null, which implies either that the volume remains constant during the transformation, or that the substance is sufficiently soft to undergo

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deformation without notable resistance, or that it is sufficiently brittle to break without deformation.

If, therefore, the final products of the transformations are soluble in each other, or if, being insoluble, the transformation is accompanied by a change of volume and the final products have sufficient cohesion to withstand a fair amount of tensile stress, the transformation will take place at a constant pressure and, consequently, at a variable temperature; it may remain incomplete.

Let us apply these propositions to the transformations of iron.

The transformation of *Gamma* into *Beta*-iron is accompanied by a change of volume, but at 860° the plasticity of pure iron is too great for the existence of much mechanical pressure. The transformation, moreover, is sudden. Therefore, *Gamma*-iron and *Beta*-iron are insoluble in each other.

Mr. Le Chatelier has shown* that the transformation of *Beta*-iron into *Alpha*-iron is not accompanied by any appreciable change of volume, at least at the normal temperature of 750° to 700°, and, moreover, the metal is still very plastic at this temperature; a strong mechanical pressure could not, therefore, be created. The transformation, however, is not sudden. *Beta*-iron and *Alpha*-iron are, therefore, soluble in each other. If mutual solubility implies isomorphism, *Beta* and *Alpha*-irons must be isomorphous.

The isomorphism of *Beta*-iron and *Alpha*-iron would also explain the possibility of variable and *permanent* equilibriums when the transformation no longer takes place at the normal temperature, but below 400°, as, for instance, in the cases of manganese and nickel steels containing suitable proportions of these elements, and in the tempering of carburetted steels. It would be, therefore, as impossible to find *Beta*-iron and *Alpha*-iron juxtaposited in hardened steel, as to find, in a piece of complex *spathic* iron ore, isolated crystals of carbonates of iron, of manganese, and of magnesium forming an isomorphous mixture.

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* *Comptes Rendus,* Vol. CXXIX, page 279; July 31, 1899. — *The Metallographist, January,* 1900, page 38.

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Morphological Relations between the Three States of Iron.

The similar structure of *Beta*-iron and *Alpha*-iron indicates the probable existence of cubic cleavages.

The cubic cleavages of *Alpha* and *Beta*-irons have been shown very clearly by Mr. Stead* in the following instances:

1. In a very soft and very brittle piece of steel (carbon, traces; phosphorus, 0.03 per cent), which had probably been heated a long time in an annealing furnace, at a relatively low temperature (see Fig. 52).

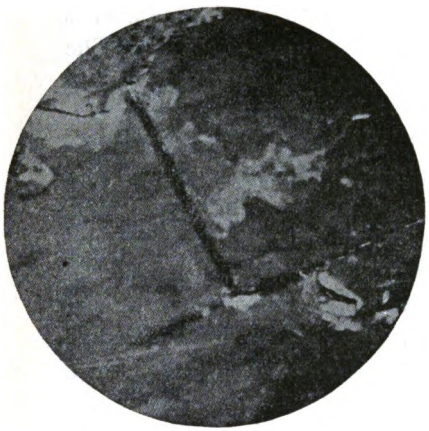

Fig. 52.

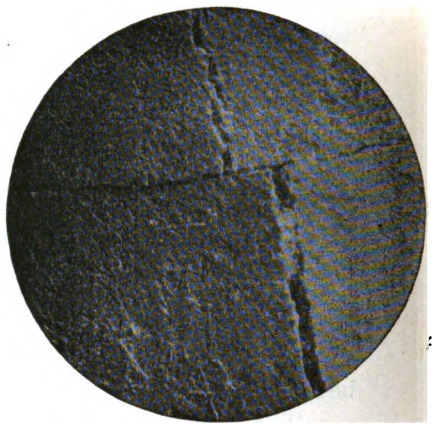

Fig. 53.

2. In a piece of iron containing 0.75 per cent of phosphorus, as stated above.

3. In reheated sheets which for some unknown reason were very brittle (Figs. 53 and 54). Since, however, in this case the orientation of the cleavages bears a certain relation to the direction of the rolling, this instance is probably a complex one.

Mr. Stead has also ascertained that the mechanical cleavages coincide with the junction lines of the small cubes revealed by the etching.†

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* *Journal Iron and Steel Institute,* 1898, I, page 178. — *The Metallographist,* Vol. I, page 289, October 1898.

† *Journal Iron and Steel Institute,* 1898, II, page 140. — *The Metallographist,* Vol. II, page 89, April 1899.

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It may be possible that the transformation of *Beta*-iron into *Alpha*-iron includes a subdivision of the cubes of *Beta*-iron into smaller cubes of *Alpha*-iron having the same orientation. At least Fig. 51 and Fig. 55, reproduced from a photograph of Mr. Stead, and especially the drawing of Mr. Andrews already mentioned, convey the impression of a *superposition* of parallel cleavages of two different orders. The existence of such a subdivision, however, does not imply that it is due to the molecular transformation; moreover, the question is possibly of little importance.

It has been seen, on the other hand, that *Gamma*-iron appears

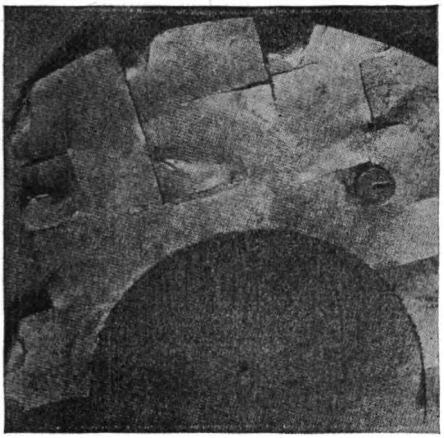

Fig. 54.

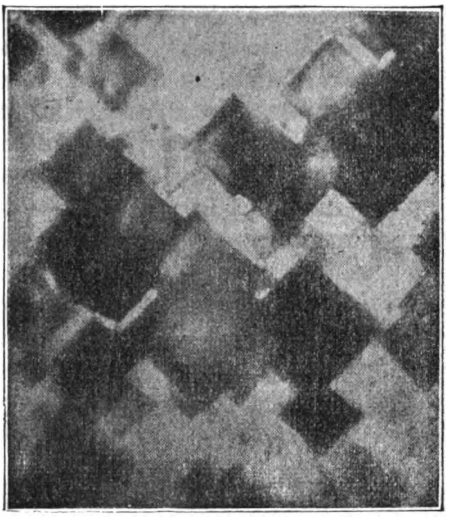

Fig. 55.

to possess, besides *octahedral* cleavages, some cubic cleavages. It may be asked, therefore, whether *Beta*-iron crystallizes around the same axes as *Gamma*-iron. The question has some bearing upon the theory of annealing, for, if such were the case, it would be difficult to explain how the structure acquired during the solidification period can be destroyed by a purely thermal treatment. The observations upon this point are somewhat contradictory.

On the one hand, Mr. Heyn has noticed, in the furnace salamander free from combined carbon already mentioned, that the small cubes revealed by the etching operation, had, at least in

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certain regions, their sides parallel to the planes of ready cleavage; owing to the nature of the sample these cleavage planes appear to be due to *Gamma*-iron unless the iron contained much phosphorus. Mr. Stead* succeeded in breaking into cubes some nearly pure iron, whose grains measured as much as one inch, and upon each of the six *unpolished* sides of these cubes the etched figures were everywhere square, with their sides parallel to the sides of the cubes. As the determinant cause of the large cleavages, however, is unknown, it is not possible to suggest an explanation.

I have, on the other hand, carefully examined a longitudinal section of Professor Tschernoff's crystal (Fig. 22), etching it by Mr. Heyn's method: the bands of ferrite, which, on the whole, are parallel to the small axes, cross each other, forming small grains in which the elementary cubes had certainly no special orientation with regard to the primitive axes of the crystal itself. It follows from this that *Beta*-iron had not borrowed the axes of *Gamma*-iron. The samples of steel cast in sand shown in Figs. 24 and 25 have yielded results which are not as plain, but which appear to me to point in the same direction.

It would follow from the above that the point A_3 causes a disturbance of the planes of symmetry, and the cubic cleavages along which it was possible to break certain samples (blast furnace salamanders, for instance), and which had appeared to belong to *Gamma*-iron, could possibly be due to *Beta* and to *Alpha*-iron.

On the other hand, however, it is quite possible that the diffusion of the carbon should exert a certain influence in the alteration of the axes at the point A_3, and that this alteration of the axes is confined to carburetted iron, to the exclusion of pure iron. It is certain that the known efficiency of the annealing operation in transforming cast steel appears to suppose, at the point A_3, a re-crystallization which has nothing in common with the previous crystallization.

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* *Journal Iron and Steel Institute,* 1898, II, page 141. — *The Metallographist,* Vol. II, page 85, April 1899.

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Conclusions.

I sum up below the conclusions to which this study has led us.

Iron, in its three states, crystallizes in the cubic system:

Gamma-iron, generally in octahedra, more or less imperfect.

Beta-iron and *Alpha*-iron, in cubes.

Beta-iron and *Alpha*-iron form isomorphous mixtures.

Gamma-iron does not form isomorphous mixtures with *Beta*-iron.

The transformation of *Gamma*-iron into *Beta*-iron appears to include a change in the planes of symmetry, at least in the carburetted irons.

Is the transformation of *Gamma*-iron into *Beta*-iron a case of dimorphism? It is simply a question of definition. It is not a case of dimorphism if the phenomenon must include a change of crystalline system; while it is a case of dimorphism if the criterion is the impossibility of crystallizing together. Mr. Wallerand* has found instances similar to that of iron, and he considers them as instances of dimorphism.

As early as 1852 Fuchs† had explained the hardening phenomenon, the residual magnetism, etc., by the dimorphism of iron; it was already the allotropic theory; it was, however, not necessary to suppose, as did Fuchs, that one of the varieties of iron, the hard variety, *Beta*-iron, was rhombohedral. Not only is there no proof, or even a beginning of proof, that such is the case, but if the views outlined above are correct, isomorphism might be the base of the mutual solubility of solids, and the mutual solubility is probably the base of the allotropic theory. The belief in the existence of a rhombohedral variety of iron, a belief which still exists, seems to have had for origin the mode of crystallization of Spiegeleisen. It was at the time thought, not without plausible reasons, that the crystals of white iron, with or without manganese, represented the allotropic variety of iron made stable at the ordinary temperature through the presence of manganese or of dissolved carbon. This opinion, however, was no longer sustained the moment it was known that the crystals of white cast iron were made up of a definite compound

* *Dingler's Polytechnisches Journal*, Vol. CXXIV, page 346, 1852.

† *Comptes Rendus*, Vol. CXXVI, page 1586, 1898.

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(Fe_3C), in which a part at least of the iron may be replaced by manganese.

The conclusions of this essay will perhaps appear artificial, and some among those of my readers who have been fed with *Novum Organum* will undoubtedly accuse me of having sacrificed to the *idola specus* and to the *idola theatri.* I admit also that more conclusive experiments are needed, and I have already planned them.

APPENDIX.

Crystallography of Cementite.

Cementite is, as we have seen, the definite carbide Fe_3C, considered as one of the mineralogical constituents of carburized iron.

Margueritte, who was apparently the first one to prepare it by synthesis in passing carbonic oxide over red-hot iron dust, did not state that his product was crystallized.* Mr. Moissan, who obtained it by quenching some cast iron which had been saturated with carbon in an electrical furnace, does not give any crystallographic description.†

In industrial carburetted iron, cementite is a deposit of second consolidation, i.e., it possesses only exceptionally its natural terminal forms.

As early as the beginning of this century Dr. Clarke found at Borrowdale some polyhedric pieces of iron, and he thought that the form of that substance was an oblique prism with a rhomboid as a base, the angle of which was 118°. His observation, however, might apply also to pseudo-crystals.‡

The only modern observation dealing with cementite is due to Mr. Stead, who found in some refined cast iron perfect elongated parallelograms.§ A section, however, taken haphazard in no special direction, does not permit any conclusion.

The double carbides of iron and manganese, of iron and chromium, of iron and tungsten have been studied more. The carbides of iron and manganese might have a bearing upon the

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* *Comptes Rendus,* Vol. LIX, page 726, October 1864.

† *Comptes Rendus,* Vol. CXXIV, page 716, April 1897.

‡ *Annales des Mines,* Vol. VIII, page 166.

§ *Journal Iron and Steel Institute,* 1898, I, page 182. — *The Metallographist,* Vol. I, 1898, page 333.

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structure of cementite because the carbide Mn_3C corresponds to the carbide Fe_3C, and because iron may be replaced, at least to a certain extent, by an equivalent quantity of manganese without producing an alteration in the chemical structure. Messrs. Carnot and Goutal, however, believe that they have isolated some definite double carbides.* Moreover, cementite, which contains little manganese and which is magnetic at the ordinary temperature, undergoes a transformation at a red heat, when it becomes non-magnetic, and may differ also from cementite containing more manganese which is non-magnetic at all temperatures.

Such being the case, we may only sum up the observations and cannot undertake to discuss them.

Professor Martens, who was the first to examine polished sections of Spiegeleisen, in most cases developing their structure by heating the sections, noted that cementite was frequently mixed with crystallites of *Gamma*-iron, or that it surrounded such crystallites, and that the crystals of cementite itself, in those regions where it was possible for them to form, were rhombic prisms, seldom possessing sharp outlines (Fig. 50, page 197).

Mallard has examined the whole series of ferro-manganeses. From 11 to 52 or 55 per cent of manganese, the crystalline form is that of a prism m of 112° 33′, modified by faces g^1 tangent to the acute angles of the prism. The faces g^1 are generally well developed, and the faces m appear only as lateral sharp edges. Sometimes the crystals form long blades, elongated in the direction of the edges $\frac{m}{m}$, and upon which the faces m are arranged in such a way as to form parallel steps.

Between 52 and 55 per cent of manganese the crystalline form undergoes a sudden change, and the structure becomes bacillar. The little parallel rods are hexagonal prisms with their faces slightly rounded. The form corresponds to that of a rhombic prism of about 120°, modified by tangent faces g^1.†

B. Rathke agrees with Mallard in the case of high percentages of manganese. According to him, however, the crystals containing 45 per cent of manganese and 6.50 per cent of carbon would be ortho-rhombic prisms of 136° 10′, truncated along the

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* *Comptes Rendus*, Vol. CXXVIII, 1899, page 209.

† *Bulletin de la Société Minéralogique*, 1879, page 47.

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acute angles by faces h^1 forming with the face *m*, the angle of about 112° which Mallard had considered as the angle of the prism.*

Professor Behrens studied some sections of alloys of iron and manganese,† but without being able to detect very precise indications concerning the crystallography of cementite.

At all events, it is certain, that cementite does not crystallize in the cubic system. If, therefore, solid solutions are isomorphous mixtures, as claimed by Mr. Le Chatelier, whose opinion is authoritative, martensite would not be, at a red heat, a solution of Fe_3C *as such,* in *Gamma*-iron. This solution would allow either graphite or cementite to be deposited according to the actual conditions of temperature and pressure. In the octahedral *Gamma*-iron, however, carbon would be dissolved in its octahedral form, i.e., as diamond; hence the brilliant synthesis suspected by Julien, anticipated by Mr. Werth and realized by Mr. Moissan.

APPLICATION OF THE LAW OF PHASES TO ALLOYS AND TO ROCKS.‡

By H. LE CHATELIER.

THE law of phases of W. Gibbs has proved a precious guide in the study of chemical equilibriums; without it, it would have been impossible to undertake the study of the complex systems which have been the object of the investigations of many Dutch scientists.

This law, moreover, may give very useful indications concerning the constitution of complex solid substances, such as metallic alloys and rocks. Their constitution is rigorously regulated by the law of phases, in all cases where the final condition is a state of equilibrium, i.e., has been reached by a series of reversible transformations. The number of different substances juxtaposited in the total mass will depend entirely upon the number of different constituents which make up its composition.

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* *Bulletin de la Société Chimique,* Vol. V, 1891, page 954.

† *Mikroskopisches Gefüge der Metalle und Legirungen,* page 130.

‡ *Comptes Rendus,* January 8, 1900.

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Let us recall the wording of the law of phases:

Let n be the number of independent constituents; p the number of physical actions; r the number of phases, i.e., of the different homogeneous substances (combination, dissolution) which are present.

The degree of liberty of such a system, that is, the number of independent variations that it may undergo, is

$$n + p - r.$$

If only one of the external physical conditions, the temperature, is variable, as is the case in the fusion of substances under atmospheric pressure, then $p = 1$, and the value of the degree of liberty becomes

$$n + 1 - r.$$

The system is said to be *invariant, monovariant, divariant* ... according to the value of the above expression, which may be 0, 1, 2 ..., etc. It means that it is possible, without disturbing the state of equilibrium, to vary 0, 1, 2 ..., etc., of the conditions which determine its actual state. These conditions are the temperature and the composition of the various phases present.

In applying this law to a mixture of solid bodies at the ordinary temperature and resulting from a series of reversible transformations, such as solidification through cooling, crystallization of a solution, etc. ..., the following conclusion is reached:

The stable state of a solid mixture (melted salts, metallic alloys, rocks, etc.) corresponds to a monovariant system; i.e., *the number of phases must be equal to the number of independent constituents which enter into its composition.*

Let us take as an instance an alloy of iron and carbon; to these two constituents must correspond two phases, either the constituents themselves, or some of their combinations. To illustrate it, let us follow the mixture from its molten condition to its condition at the ordinary temperature.

1. *Liquid cast iron.* — 1 phase; divariant system; the temperature and composition of the liquid are independent.

2. *Liquid cast iron and graphite.* — 2 phases; monovariant system. The composition of the liquid is constant at each temperature. It is a saturated solution.

3. *Liquid cast iron, graphite, solid carbon solution.* — 3 phases; invariant system. The composition of the iron, 3 per cent

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carbon, that of the solid solution, 1.5 per cent carbon, and the temperature, about 1150° C., remain constant as long as one of the three phases has not disappeared. Upon further cooling the liquid phase disappears.

4. *Graphite, solid carbon solution.* — 2 phases; monovariant system. The composition of the solid solutions (austenite, martensite, or troostite) is constant at each temperature. It is a saturated solid solution.

5. *Graphite, solid carbon solution, pure iron.* — 3 phases; invariant system. The composition of the solid solution, about 0.5 per cent of carbon, and the temperature, about 700° C., remain constant as long as none of the 3 phases disappears. In continuing to abstract heat, the solid solution disappears.

6. *Graphite and pure iron.* — 2 phases; monovariant system. The temperature may vary without altering the condition of the system, which is retained at the ordinary temperature and is then absolutely stable.

The result will always be the same in substances obtained through fusion. The cooling of an invariant system below its point of last solidification is always accompanied by the disappearance of one of the phases. The system becomes monovariant and remains so to the ordinary temperature, provided none of the solid substances undergo any subsequent transformations during the cooling. Otherwise, it passes through a new invariant point, as in the case of carburetted iron, and always reaches finally a monovariant system.

As an instance of substances containing three constituents which must, after solidification, contain three juxtaposited phases, granite may be given. Its three elementary constituents are silica, alumina and oxide of potassium; its three phases are quartz, feldspar and mica.

If, in such a system, a number of phases is found superior to that of its independent constituents, it is certain that the series of transformations from which they result is not reversible, and that the final condition is not stable.

We find an instance of this in the case of slowly cooled cast iron, which is generally made up by the juxtaposition of three substances: iron, graphite and cementite (Fe_3C). Such a state is unstable. In reheating (manufacture of malleable cast iron) the cementite disappears.

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The two principal causes which produce these unstable systems are, first, too rapid cooling, as in the case of cast iron, and, secondly, the successive consolidation of the various elements crystallizing in spherical layers, resulting in the complete isolation of certain parts. This explains why three phases are frequently found in alloys of copper and cadmium, namely, copper, cadmium and a combination of the two metals. Some of the copper which solidified first is surrounded by the definite compound and thus prevented from further combining with the cadmium.

An important attribute of these unstable alloys is to possess a constitution, and therefore physical properties, varying greatly with the working conditions by which they have been produced.

IRON AND STEEL FROM THE POINT OF VIEW OF THE "PHASE-DOCTRINE."*

By BAKHUIS ROOZEBOOM.

IT is a great honor for me to present to the Iron and Steel Institute a summary of a recent article of mine under the above title,† at the request of your President, Sir W. C. Roberts-Austen, whose eminent researches form, in fact, the basis for my theoretical study.

My guide in this study is the phase-doctrine of Gibb. I think as yet this is very little known by most of the members of this Institute, but it is, nevertheless, a conception which has already proved its usefulness in the explanation of many an entangled chemical problem. One of the chief objects of this inquiry is to find out the conditions of the existence of the phases of a system of one or more components. In applying it to iron and steel, we have a system of two components — iron and carbon; and by the phases of this system we understand all the distinct forms which may appear in the system. We have, therefore, to consider the following phases: carbon, iron, which, if we accept the allotropic theory, may present itself in three phases — *Gamma, Beta,*

* Iron and Steel Institute, Paris Meeting, September 1900.

† *Zeitschrift für Physikalische Chemie,* Vol. XXXIV, 1900.

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and *Alpha;* liquid solution, solid solution of carbon in *Gamma*-iron or martensite; cementite or carbide, Fe_3C. Thus it will be evident that a " phase " may be liquid or solid, may be an element or a compound, or a homogeneous mixture of variable concentration. But " pearlyte " does not represent such a phase, for it is a conglomerate of two solid phases, ferrite or *Alpha*-iron and cementite.

Now it has been deduced from thermo-dynamical principles that there are only a very limited number of ways in which a liquid phase formed by two components may solidify to one or more solid phases; and this being so, there are a limited number of ways in which a transformation in or between these solid phases may take place. Furthermore, the application of the phase-doctrine is so general, that it matters not whether we start from a liquid phase built up from water and a salt, or from two salts fused together, or from two silicates, or from two metals, or from iron and carbon. Therefore the point of view of the phase-doctrine embraces widely different problems.

I have, therefore, examined the problem of iron and steel from this point of view, making use of the most accurate numbers obtained by Sir W. Roberts-Austen in his recent studies. Fig 2 represents the results of my study. It resembles in many respects Fig. 1, which is Roberts-Austen's. In fact, the new lines that are introduced principally result from the knowledge I had obtained as to the solidification and transformation of solid solutions, which enabled me to give a better correlation of the different parts of the figure.

Let us consider first the solidification of liquid solutions with 0—2 per cent carbon. I have added the curve A*pra*, which has been traced as a straight line, because its position is not exactly known. All these solutions with 0—2 per cent of carbon solidify to homogeneous solid solutions, but the process takes place in the following manner:

Take a solution *q*, trace the horizontal line *qp*, then *p* represents the composition of the first crystals that separate from that solution. They *must* contain less carbon than the liquid. The liquid will be enriched in carbon, and its temperature of solidification is thereby lowered, and so the liquid passes successively from *q* to *s*. In the meanwhile the mixed crystals of iron and carbon change also with the fall of the temperature from *p*

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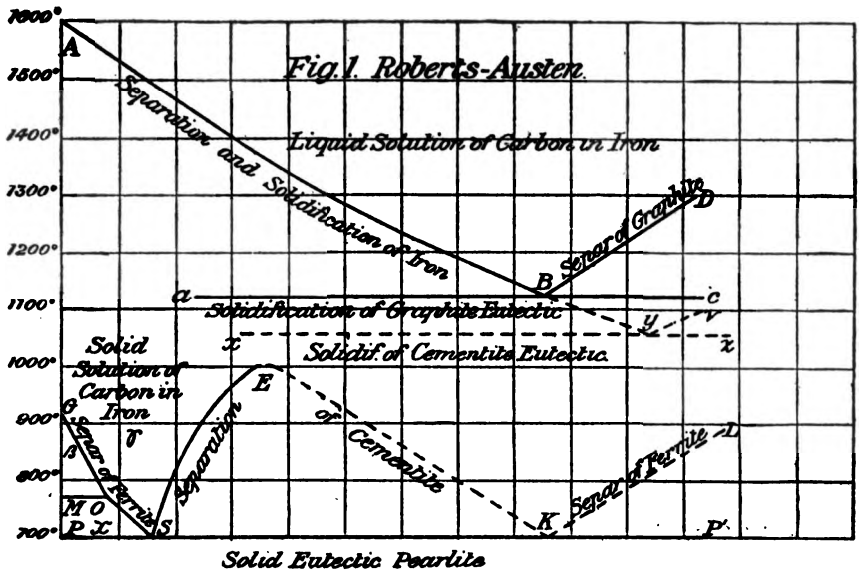

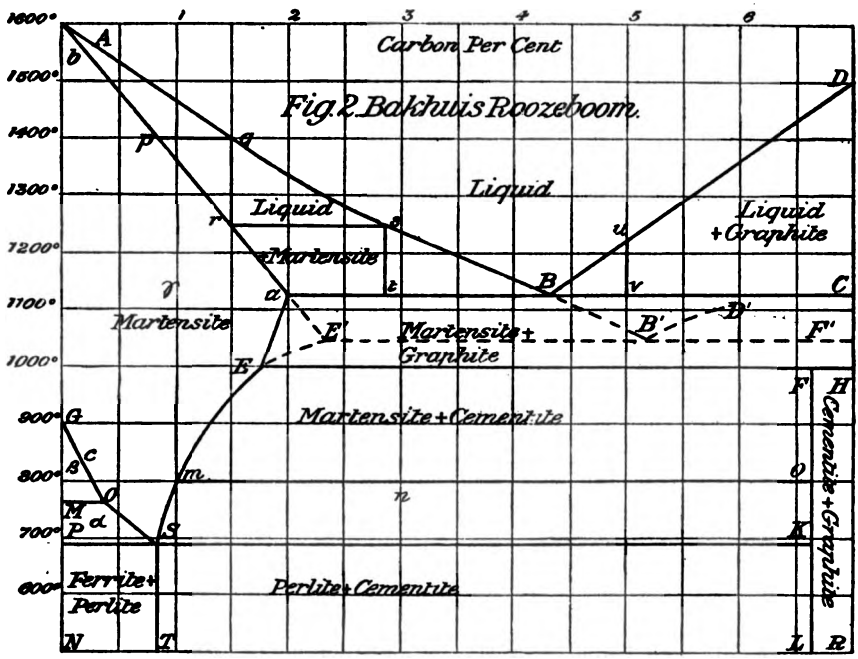

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to *r*, and at last the whole solution becomes solidified into homogeneous mixed crystals of the composition *r*, that of the original liquid being *q*. The solidification, however, has not taken place at a constant temperature, but over the interval *qr*.

So it proceeds until the point *a* is reached, which indicates the presence of 2 per cent of carbon. From 2—4.3 per cent of carbon there is first a gradual solidification as before, until the remaining liquid has reached the point B, and the crystals have reached the point *a*, and then the remaining liquid solidifies at a constant temperature of 1130°, according to Roberts-Austen, so as to form what I like to call a conglomerate of carbon and mixed crystals of the composition *a*, which may perhaps be 2 per cent of carbon.

At 1130° there exists by contact with one another a liquid solution of 4.3 per cent of solid carbon (graphite), and a solid solution (martensite) containing 2 per cent of carbon. Three phases are the maximum that may coexist, according to the phase-doctrine, in the case of stable equilibrium at a constant temperature.

Turning to the field above the curve AB, all its points represent liquid solutions at various temperatures and concentrations; therefore it may be called the field of the liquid phase. In the same way the points beneath A*a* will represent different solid solutions, each of which may exist undisturbed by the fall of temperature so long as a vertical line denoting their composition does not encounter a new curve.

In the case of the field A*a*B it is, however, otherwise. A point in the field does *not* represent a homogeneous phase, but a complex of two phases. Take a point on the line *pq;* then at this temperature we have seen that there can exist in contact with each other the liquid phase *q* and the solid solution *p;* a liquid with less carbon than *q*, and a solid with more carbon than *p* are both impossible at the given temperature; therefore, a point between *p* and *q* can only represent a complex of a certain proportion of the liquid phase *q* and another of the solid *p*. The nearer the point to *p*, the greater is the proportion of the solid solution — and conversely. What condition will be attained at a given temperature will depend on the concentration of the initial liquid before solidification began.

It will be the same in the other fields, in which the names of

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two phases are inscribed in Fig. 2. Their points always represent a complex of two phases which are to be found at the ends of a horizontal line drawn through the field. So the point *n* represents a conglomerate of martensite of the composition *m* and cementite *o*, the whole perpendicular LF indicating cementite, which is a compound of definite composition at all temperatures.

Liquid solutions with more than 4.3 per cent of carbon deposit graphite as long as they traverse the field from BD to BC.* The remaining liquid descends from D to B and solidifies totally at 1130° to martensite and graphite.

We will now first trace the history of the mixed crystals which can exist beneath the curve A*a*. I have changed nothing in the Roberts-Austen lines GO, OS, and SE, and the interpretation of the separation of *Beta*-iron, *Alpha*-iron, or cementite is also accepted. That a solid solution may reject one of its components or a compound just as a liquid solution does when it is cooled, was an entirely unknown phenomenon a few years ago, but it has now been demonstrated in some cases which have been the object of researches by my pupils.

The existence of three allotropic states of the iron, the discovery of which we owe to Mr. Osmond, is not exceptional. The researches of later years have so multiplied our knowledge of the allotropic forms of solid bodies, that it is at present far more peculiar for a body not to present allotropy than to do so. As regards metals, there is the new case of tin, which presents a transition point at 20° C., just like the transition point of *Gamma*- into *Beta*-iron at 890°. The succession of three such states in iron is no longer to be wondered at, for ammonium nitrate shows four of them, and therefore the transformations of the martensite crystals with 0—0.86 per cent of carbon only admits of a rational explanation if the existence of the three varieties of iron is admitted. The right understanding of these phenomena was, however, impeded by the fact that no analogous phenomena were known to occur at more convenient temperatures. I have, therefore, pointed out the complete analogy of the lines GO and OS with the lines that represent the successive separation of the dif-

* Two questions arise here: 1st. Whether the graphite which is separated is pure carbon, or whether it is also a solid solution (of iron in carbon)? 2d. Whether the graphite that separates retains the same form until its fusing-point?

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ferent modifications of ammonium nitrate from its liquid solution, according to the amount of water present. The same phenomena are observed in the separation of solid $TlNO_3$ or $AgNO_3$ from their fused mixture.

My studies on mixed crystals led me, however, to consider also the possibility that the *Beta*-iron and *Alpha*-iron also hold some carbon in solid solution, although less than the *Gamma*-iron. This would give some complications in the figure GOSP which it is difficult to describe briefly. It would, however, greatly influence the results obtained in an attempt to deduce the molecular weight of the carbon in the solid solution from the observed lowering of the transition point.

As regards the iron-carbon alloys with more than 2 per cent of carbon, my results are more at variance with those of Sir W. Roberts-Austen. Beneath the line *a*BC at 1130° in the normal case there should be nothing but a solid conglomerate of two phases, viz., graphite and martensite with 2 per cent of carbon. Firstly, I have traced the line *a*E to indicate that the amount of carbon in the martensite falls simultaneously with the temperature, and this would induce a further separation of graphite. Then at about 1000° the line *a*E meets the cementite line SE of Roberts-Austen. This can have no other meaning than that at 1000° there is in the normal state of stable equilibrium an abrupt transformation of the graphite into carbide (cementite), according to the equation —

Martensite 1.8 per cent. C. + Graphite = Cementite (Fe_3C).

I have sought for analogies to illustrate this peculiar transformation. There are as yet only two remote analogies, both of which were discovered in my laboratory — the transformation of a solid solution of *d* and *l* camphor-oxine in the racemic compound, and the transformation of a solid solution of HgI_2 and AgI_2 into the compound HgI_2, 2AgI. They are the very first examples in which a transformation from a solid solution into a compound has been studied. Both have, however, the peculiarity that at higher temperatures the series of mixed crystals that can exist, embraces also the ratio in which the components form the compound. There is, on the contrary, no solid solution possible above 1000° with a percentage of carbon equal to that in Fe_3C. Therefore we have a type of the second method of formation of

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compounds such as presents itself very often in liquid salt solutions; for instance —

Salt solution + anhydrous salt = hydrate of salt.

The formation of iron carbide from martensite and graphite is doubtless the analogous formation of a compound from a solid solution. But then the temperature of 1000° is a transition point; that is, only at that temperature can the three phases coexist, and, therefore, the transformation of all the conglomerates of martensite and graphite must take place at this temperature; the border line EFH must be horizontal.

It is impossible to state briefly here all the arguments which have led me to accept this line in preference to the lines EK and KL of Roberts-Austen and of Stansfield; nor can I sum up the considerations which I have adduced to explain the many anomalous results obtained with highly carburized iron. A prominent part is played by the retardations which are possible in all the processes of transformation which occur during cooling. There is also to be taken into account the influence which retardation has upon the production of cementite during the period of solidification, and during the two following periods above and below 1000°.

Again, the straight line PSK at 690° is also wholly in harmony with the known interpretation that it gives the transformation of the residual solid solutions of 0.85 per cent of carbon (point S) into a conglomerate of *Alpha*-ferrite and cementite, which is known by the name of pearlyte. To the right of S this same transformation is accomplished at the same temperature in martensite which is accompanied by cementite; to the left of S it is accomplished in martensite accompanied by *Alpha*-ferrite. Therefore 690° is also a transition point.

As to the possibility of passing this point as well as the curves GO, OS, ES, in abrupt cooling, and thereby preserving to the ordinary temperature the *Gamma*-state of the iron as mixed crystals or solid solutions, I have nothing to add to this way of explaining the process of hardening.

The different fields obtained in Fig. 2 give the limits of concentration and temperature for the most prominent of the phases that appear in the carbon-iron system. There are some others about which our knowledge is far too imperfect to make it pos-

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sible to indicate their place. Only in the case of austenite has such an attempt been made, but the position austenite should occupy in the system still remains highly hypothetical.

The previous endeavors to determine the molecular weight of carbon in the solid solution may be re-examined in connection with the new line A*a*, and possible alterations in the figure GOM. The results of such an examination point to the fact that the carbon molecule probably only contains a single atom, but it cannot be decided whether the carbon is free or is present as Fe_3C.

THE PRESENT POSITION OF THE SOLUTION THEORY OF CARBURIZED IRON.*

Part II.

By A. STANSFIELD.

Introduction.

IN my paper at the Autumn meeting in 1899, an outline only of the solution theory of carburized iron, in which our President is so much interested, was presented to the Institution, and no attempt was made to reconcile with this theory the conflicting opinions and statements which have been made from time to time. In the present paper, which may be regarded as a continuation of the first, I propose to review as far as possible all the experimental evidence that appears to bear upon the subject, and also to incorporate the results of some fresh experiments of my own.

In view, however, of the magnitude of such a task, I shall limit the present paper to a consideration of a part only of the whole subject; the part selected being the conditions under which graphite and cementite are formed in carburized iron.

The great interest of the relations existing between carbon and iron has attracted the attention of no less an authority than Professor Bakhuis Roozeboom, whose knowledge of the analogous cases of fused mixtures of salts and of saline solutions generally,

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* Iron and Steel Institute, Paris Meeting, September 1900. — For Part I, see *The Metallographist*, Vol. III, page 24, January 1900.

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gives great value to the paper he has just written: "Eisen und Stahl vom Standpunkte der Phasenlehre."* No apology will, therefore, be needed if, in treating the subject, I attempt to meet some of the questions which he considers demand immediate attention, and in doing so I can refer to those of his diagrams which will be reproduced in the summary which he is communicating to the Institute.

In discussing the formation of graphite and cementite, it will be advisable to begin with the molten material, and to trace the changes which occur as it cools.

The Solidification of Carburized Iron.

This, as was stated in the first paper, is now generally admitted to be closely analogous to the freezing of a saline solution, the solids which separate being: — 1. Iron (in the *Gamma*-state), containing a variable amount of carbon dissolved in it; 2. graphite; and 3. a eutectic or fusible mechanical mixture of 1 and 2.

The amount of carbon which is held in the state of solid solution in the iron depends upon the amount of carbon in the fluid solution from which it separated, and, until the saturation point is reached, it is probably roughly proportional to it. Professor Roozeboom has recognized this fact by the addition, in Fig. 2 of his paper, of the line A*a*, which represents the proportion of dissolved carbon in the solid iron which separates at any temperature, and this line, and other analogous lines which will be found in this paper, enable the very complicated facts to be more readily interpreted.

The line A*a* is drawn from A, the freezing-point of pure iron, to a point *a* on the eutectic line *a*BC; the position of *a* denoting the maximum amount of carbon which solid iron can permanently hold at that temperature without separation of graphite. The exact position of *a* has not yet been determined, but it could probably be obtained experimentally by cooling a series of pure carburized irons very slowly from the molten condition to just below 1130° and then quickly to the ordinary temperature: the proportion of carbon for which graphite just began to separate, would indicate the true position of *a*.

In my last paper I placed *a* at 1.2 per cent carbon, as this was

* *Zeitschrift für Physikalische Chemie,* Vol. XXXIV, 1900, page 437.

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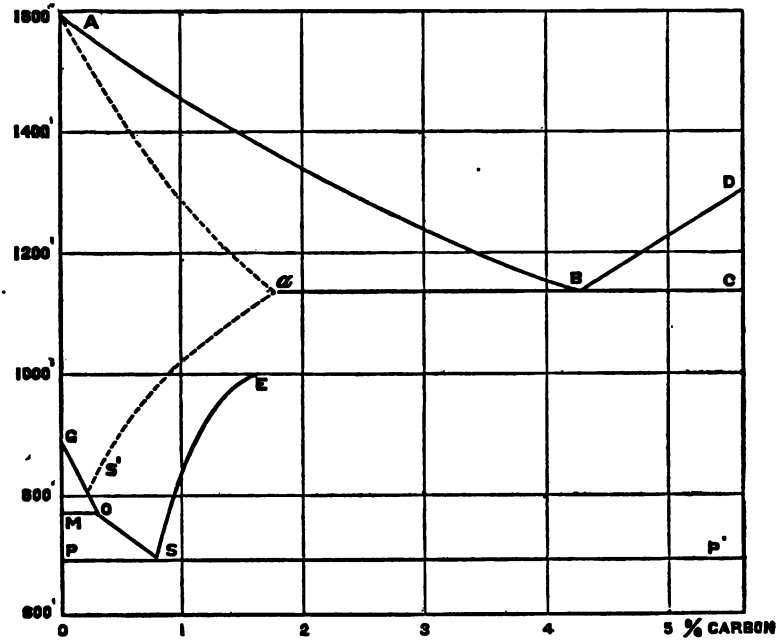
the point at which the formation of graphite was evident in the cooling curves. A careful consideration of the progress of solidification as described by Professor Roozeboom in his summary, will bring to light the important fact that the process can only take place as he describes it *if the rate of cooling is very slow.* Thus in his Fig. 2, tracing the solidification of a liquid having the composition *q*, the solid which separates will have at first the composition *p*, but as the composition (and temperature) of the re-



sidual liquid changes from *q* to *s*, that of the deposited solid will have changed from *p* to *r*. Now we know that when solid crystals are formed in a solution, it is the solution in *immediate contact* with the crystals which is at first impoverished, but, owing to the rapid mixing and diffusion which takes place in liquids, each part of the solution is kept at approximately the same degree of concentration as the rest. In the case of solids, however, it is obvious that no mechanical mixing can take place, and the rate of diffusion in solids has been shown to be much less than in liquids, and this must obviously be the case. It will be evident, therefore,

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that if in the solution *q* a crystal of steel (that is, iron with carbon dissolved in it) is formed having the composition *p*, and if subsequent growths of the crystal have compositions varying from *p* to *r*, we have by the time the residual liquid has reached the composition *s*, not a uniform crystal of composition *r*, but a crystal of composition *p* at the centre and *r* at the outside. The *average* composition of the crystal will be between *p* and *r*, and there will, therefore, be some of the enriched liquids remaining, which may eventually form a eutectic. It will be evident, therefore, from this that the process of solidification will not in general stop at the line A*a*, but that the formation of the eutectic alloy, with subsequent separation of graphite, will take place in steels containing a smaller percentage of carbon than corresponds to the point *a* (which is about 1½ per cent or 2 per cent). In proportion as the rate of cooling is decreased, the diffusion of carbon in the solid steel crystals will make the whole approximate more and more to the composition *r*, and when equilibrium has been established, it will coincide with *r*, and the whole liquid will consequently have solidified, a fact which is capable of easy arithmetical demonstration. In a true equilibrium curve, which Professor Roozeboom's Fig. 2 really is, the eutectic line B*a* will stop where it meets the line A*a*.

In view of the above, it is probable that the position of *a* will be somewhat to the right of that given in my last paper,* which was 1.2 per cent carbon. The position assigned to it by Professor Roozeboom (2 per cent) is probably approximately correct. In my last paper I advanced the hypothesis, based on the slope of the line AB, that the carbon in fluid iron was monatomic, and that the iron which solidified contained about half as much carbon as the liquid from which it was formed. This would give to *a* the composition 2.1 per cent carbon, which is in general agreement with the 2 per cent suggested by Professor Roozeboom. The monatomic condition of carbon in molten iron is, moreover, supported by the observation of Ramsay that the metals in fluid alloys are monatomic, carbon here playing the part of a metal.

The following equation, which is a modification of that of Le Chatelier, enables the exact shape of the line A*a* to be deduced from that of the line AB, provided that the latent heat of fusion

* *Journal Iron and Steel Institute*, 1899, II, page 174.—*The Metallographist*, Vol. III, page 24, January 1900.

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of pure iron be known. The assumption is also made that the carbon is monatomic in the liquid metal. In a somewhat abbreviated form, which will be sufficiently accurate for the present purpose, the equation is $2\frac{dx}{x}(1-c) = \frac{\lambda}{T^2}dT$. In this equation x is the number of molecules of iron in 100 molecules of iron and of carbon in the molten alloy. T is the temperature of solidification measured from the absolute zero (— 273° C.), and c is the ratio between the percentage of carbon in the solid and in the liquid from which it was formed, the percentage being reckoned in molecules. λ is the latent heat of fusion of one gram molecule of iron. Taking Person's value of 20 calories per gram, it becomes 1120.*

The result of this calculation places *a* at about 2 per cent, but I have adopted 1.8 per cent provisionally, as it appears to agree better with the evidence obtained from other sources. The general curvature of the line A*a* is, however, obtained by means of the calculation; it will be observed that it has a somewhat stronger curvature than the line AB.

Turning now to the solidification of liquids to the right of B, the process is the same as has already been described, except that a eutectic alloy is finally left containing about 4.3 per cent of carbon. This solidifies at points cn the line *a*B, forming a mechanical mixture of graphite and a solid solution of carbon in iron having the composition *a*. I may incidentally observe that the true meaning of a eutectic alloy has already been fully described and need not be dealt with here.

Passing to the right of B, an excess of carbon is found above the amount required to make the eutectic, which contains 4.3 per cent of carbon. This excess of carbon crystallizes out as graphite at points on the line BD. Professor Roozeboom asks whether this graphite is pure carbon, or whether it too is a solid solution of iron in carbon. In order to test this I intend to determine the amount of iron in a quantity of "kish" which separated from a rich sample of carburized iron during slow cooling from the molten state. The kish had previously been boiled with dilute hydrochloric acid to extract any iron that might be mechanically entangled.

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* Roberts-Austen and Stansfield, *Bull. Phys. Congress,* Paris, 1900.

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There will still, however, be some uncertainty whether the iron was really dissolved or only mechanically mixed with the graphite. The question is not of great importance, but if a series of determinations of the amount of iron in graphite were found to give concordant results it would appear to be in favor of Professor Roozeboom's suggestion.

With regard to the slope of the line BD, it must be remembered that this part of the curve is somewhat difficult to obtain by the ordinary method of taking cooling curves, as the heat evolved by the separation of graphite is very slight, and the extrusion of graphite from the cooling mass renders the carbon percentage of the original fluid metal somewhat uncertain. If the line BD is prolonged up to the point which would represent pure carbon, it will be found to reach a temperature of at least 8000° C. We do not know at what temperature graphite melts, as Moissan has shown that it volatilizes without any sign of fusion at the highest temperature of the arc, say about 3000° C., but its melting-point can scarcely be so high as 8000° C. This is, however, not at all anomalous, as it is in agreement with the fact that the ideal curve is not in general a straight line, but has one or two points of curvature which would probably reduce the melting-point of graphite to a more reasonable temperature.

Professor Roozeboom's question whether there is any change in the character of the separated solid as we proceed up the line BD, must, I think, be answered substantially in the negative, as the tendency would be for the solid to become more and more nearly pure carbon, and it is practically pure to begin with; we know, moreover, of no decided change in the physical nature of graphite until it reaches the temperature of the arc, at which it rapidly vaporizes.

Changes after Solidification.

In the lower part of the diagram, Fig. 1, relating to solidified cast iron, it will be seen that important alterations have been made since my previous paper. The difficulty in obtaining these curves, by the usual method of taking cooling curves, has arisen from the extreme slowness with which the changes occur in the solid metal, and it may be well to give a short account of the different stages by which our present knowledge of the subject has been gained. When the diagram was first published, in

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1897,* the curve SE, which shows the solubility of cementite in pig iron, was left incomplete at E, as the positions of points beyond this were uncertain. The solution theory appeared to demand that the line should continue horizontally from E (as Prof. Roozeboom has drawn it), but this would have indicated that the amount of cementite in cast iron should either remain constant or should increase, with further additions of carbon; and while we know that this does occur to a certain extent, as in the case of white pig, still further additions of carbon, especially when the iron is subjected to very slow cooling, are known to result in a marked reduction in the proportion of cementite, a fact which is not consistent with the line EF in Professor Roozeboom's Fig. 2 being horizontal, except under special conditions to be described later. Up to this point the evidence had appeared to support the hypothesis that cementite was a stable body at temperature below about 1050°, at which temperature experiments had shown that it dissociated into iron and graphite. Professor Roozeboom's curve shows that the hypothesis which had been tacitly accepted is not in accordance with experimental facts. He shows that the hypothesis necessitates the assumption that *all* the graphite which is liberated as the iron solidifies, should at 1050° combine to form cementite, and that if we consider more and more richly carburized iron this process should go on until at 6.6 per cent of carbon the solidified mass, if cooled sufficiently slowly, would consist of nothing but cementite; while it is a matter of common experience that, under these conditions, there would be a large amount of graphite, and but little cementite. It follows from this that at the temperatures under consideration cementite is not really a stable body.

The fact that slowly cooled iron, when rich in carbon, contained very little carbon in the combined state has long been known in the case of pig irons, in which, however, the simultaneous presence of silicon somewhat complicates the problem. Perhaps the earliest experiment in which pure materials were used was one described by Dr. Percy,† in which he melted pure iron in crucibles brasqued with lamp-black, and obtained a cast iron containing a little more than 4 per cent of graphite, and with

* Roberts-Austen, Fourth Report to Alloys Research Committee.—*Proc. Inst. Mech. Eng.*, 1897.

† Percy's *Metallurgy, Iron and Steel*, 1864, page 113.

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no sensible amount of combined carbon. Later experiments, showing practically the same thing, can be found described in the Journal of this Institution, and some experiments of my own on the effect of prolonged annealing on carbonized iron, which in the main agree with those of Dr. Percy, will be described a little later, as they appear to raise fresh points for discussion.

The results of these experiments and of the well-known behavior of richly carbonized iron can only lead us to the belief that not only does the graphite *not* combine with the iron on slowly cooling to 1050°, but that the 2 per cent of carbon which the solid iron at first contained is rejected as graphite and not as cementite, if the metal is cooled sufficiently slowly. In other words, we are confronted with the highly important fact that carbon is more soluble in iron when it is presented to it as cementite, than when it is presented as graphite. Several analogous cases are known among aqueous saline solutions, in which the dissolved salt can separate in two or more states; either anhydrous (corresponding to the graphitic state of carbon), or containing water of crystallization (corresponding to Fe_3C). In some of these cases either form of the salt can be made to separate at will from the solution by placing in it a crystal of the desired variety, and this probably throws light upon the conditions under which graphite or cementite separates. Normally, graphite should separate, being less soluble than cementite, but, as was explained in my last paper, graphite is very bulky, and when it is remembered that the mass from which it separates is solid, it will be evident that its separation from the solid will be opposed by a very considerable force: cementite, therefore, and not graphite, is usually found in steel. If, however, flakes of graphite have separated on solidification, as in grey pig iron, these present many nuclei, around which more graphite can form, and thus relieve the mechanical pressure.

Professor Roozeboom in his paper takes the "Phase-Rule" of Gibb as his guide, and it may be well to consider the subject from this point of view. The phase-rule lays down in effect that in a system such as that of the carburized irons, in which two distinct substances (carbon and iron) are involved, but in which certain forms or phases of carbon or iron, or carbon-iron solution, or carbon-iron compound, are present, no more than two

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of these phases can exist in equilibrium with each other except at a particular temperature.

In the case of a solution of salt in water, this would mean that you could only have salt and ice and solution together at a particular temperature (the eutectic temperature), and that at any other temperature you could only have ice and solution or salt and solution (at temperatures above the eutectic), or ice and salt (at temperatures below the eutectic). In the case of a salt solution this is quite evident, but the value of the phase-rule is that we can apply it with equal confidence to cases where we do not, to begin with, know the answer to our question.

Applying the rule to the case of solid carburized iron at temperatures above that of all the known allotropic changes — we have the four possible substances of iron, graphite, cementite, and solid solution of carbon (either graphite or cementite) in pig iron. The rule tells us that only two of these can in general exist permanently together.

If we have a solid solution of carbon in iron any excess of carbon must be *either* as cementite or as graphite; we cannot have both together. We do, of course, find all three substances in the same piece of metal, but we may feel assured that they are only there because they have not had sufficient time or opportunity for effecting the desired transformation. This alternate possibility can easily be observed practically by thoroughly annealing steel or cast iron of varying degrees of carburization. There will be found to be either practically no graphite or practically no cementite. My own experiments show this very well, although the samples were only annealed for a couple of days (Fig. 1).

If then we must choose between graphite and cementite, which must we select, and why is it that sometimes one and sometimes the other appears? The answer is that graphite and not cementite is the stable form; this being shown by the experiment of annealing a cast iron containing originally both cementite and graphite.

The absence of graphite in most steel, even when annealed, arises from the fact that under the ordinary conditions (which probably include a considerable mechanical opposition to the formation of graphite), a solid solution of carbon in iron does not readily deposit graphite even when it is considerably super-

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saturated with respect to it, but that cementite will readily separate as soon as its saturation-point is reached. It will be remembered that in the case of the salt solution three phases (salt, ice, and solution) could exist together at one particular temperature. In Professor Roozeboom's Fig. 2, the analogous temperature at which cementite, graphite, and solution could exist together was represented by the point E, at which the solubility curve of cementite SE, met that of graphite *a*E. If, however, as I think we must admit, the solubility of graphite is less than that of cementite, throughout the range of temperature under consideration (900 to 1130), and in the absence of mechanical pressure, the curves cannot meet, and there can be no temperature at which graphite, cementite, and solid solution of carbon in iron can be in equilibrium. The curve of solubility of graphite in iron has been drawn provisionally as *a*S', because it must pass through *a*, and the experiments alluded to above suggest that, at somewhat lower temperatures, it approaches closely to the line, AG, of pure iron. As regards the general shape of the curve it is drawn roughly in accordance with the equation of Le Chatelier, and I believe he has already suggested that the curve should occupy some such position. It will be evident from what has already been said that the position of this curve could not be detected by means of the ordinary cooling curves, but it is to be hoped that suitable experiments will be made to obtain its correct position and that of other parts of the diagram which are at present somewhat uncertain.

Equilibrium Curves.

When the evidence with regard to the fusibility of metallic alloys was first obtained by means of a series of cooling curves, the point or points of solidification of each alloy were plotted on a diagram which summarized the results of the whole series of cooling curves, and which was known as a freezing-point curve. The term was retained for a while in connection with the carbon-iron series, although many of the points there represented denoted changes occurring after solidification. A more comprehensive term was therefore needed, and I think that the term "equilibrium curve," which is increasingly used by physicists, will be found to meet the case. Not only does the new term include the changes that occur after solidification, but it also

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tends to give greater precision to our views with respect to these changes. Thus a freezing-point, especially in the case of a solid

Fig. 1. Pure carburized iron annealed for two days. Portion near edge × 56 D.

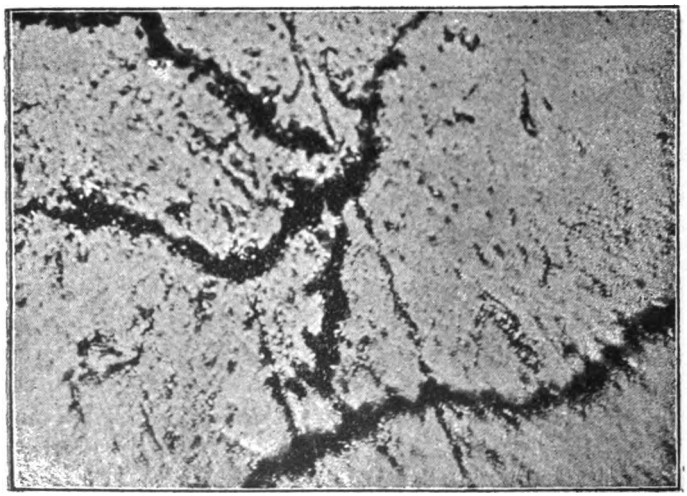

Fig. 2. Lower portion of Fig. 1 × 1580 D.

solution, may vary in position, according to the degree of rapidity of the cooling; but a point of equilibrium shows the temperature

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and composition of a solution which is in permanent equilibrium with a certain solid — that is, no matter how long it is left, the amount of solid will neither increase nor decrease. The horizontal eutectic lines cannot be defined quite so simply, and have been omitted by some physicists; they are, however, an essential part of the system of curves, and connect together portions of the true equilibrium curves, which would otherwise be isolated. It will be evident, then, that in the case of the carburized iron series, we have two distinct systems of equilibrium curves: 1. that in which carbon is present in the form of graphite, and 2. that in which carbon is present in the form of cementite. We are acquainted so far with a part only of each system, namely, with the solidification of carburized iron on the graphite system, and with the allotropic changes on the cementite system; the converse — the solidification on the cementite system (forming white pig, and in some cases, possibly diamonds), and the allotropic changes of the graphite system (that is in very slowly cooled graphitic pig) — has yet to be studied.

The dotted lines occurring in Professor Roozeboom's Fig. 2 will probably represent the solidification of white cast iron — that is, they are parts of the cementite equilibrium curves, graphite being prevented from forming by pressure or by sudden cooling. A part at least of the horizontal line, EF, probably represents the separation of cementite at a constant temperature, from the solid solution of constant composition produced by the solidification of the graphite-iron eutectic, when sufficient time for the further separation of graphite is not allowed.

Plates I. and II. show the result of two of the experiments I made on the effect of prolonged annealing on carburized iron.

The iron was carburized by melting in a graphite crucible, packed in charcoal powder, and was subsequently annealed for nearly two days at a temperature below that of its melting-point, followed by very slow cooling.

Fig. 1 is the appearance under a low power objective and represents with a magnification of 56 diameters a portion near the edge of the specimen. It will be observed that the lower part of the figure, which represents the superficial portion of the specimen, has a structure which is entirely different from the rest. A photograph of this portion under a higher power (1580 diameters), Fig. 2, shows ferrite with veins of graphite run-

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ning through it. The inner portion appears in Fig. 1 to contain a considerable proportion of segregated cementite. A rough determination of the combined carbon by color showed only about 0.4 per cent, and under the high power objective this portion (Fig. 3) appears to consist almost entirely of a badly developed variety of pearlyte, with a little graphite and cementite. How can the different structures in the two portions of the metal be explained? It can scarcely be that the outer portion has been decarburized by cementation in charcoal powder, neither can we suppose that much more carbon has entered by cementation, seeing that it had already been fully carburized by melting in contact with charcoal. The explanation which appears to me to be the most probable one is, that the mechanical pressure which opposes the formation of graphite in the body of the specimen is considerably less near the surface; graphite is consequently able to separate, and drains the combined carbon facing the surrounding iron, leaving almost pure ferrite. It is important to remember that the state of equilibrium of two or more bodies is affected by the pressure to which they are subjected, as well as by the temperature. We should, strictly speaking, have a succession of equilibrium curves, each corresponding to a particular pressure. The curves of Professor Roozeboom may well correspond to the true equilibrium of carburized iron when subjected to a considerable mechanical pressure, and that of Roberts-Austen to the somewhat mixed conditions which obtain in practice when both the temperature and the internal mechanical pressure are variable.

Summary.

The general conclusions to be drawn from the inquiry appear to be: —

1. That carbon is less soluble in iron when presented in the form of graphite than when presented in the form of cementite.

2. That the apparent reversal of this in steel is due partly to the absence of nuclei of graphite on which further deposits might take place; partly to the length of time required for the separation of the graphite, involving, as it does, the gradual passage of carbon through the iron to reach the nuclei; and partly to the mechanical pressure which must oppose the formation of graphite in solid steel.

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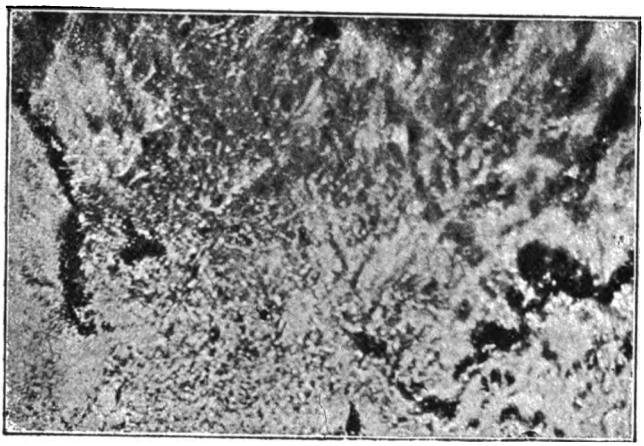

Fig. 3. Upper portion of Fig. 1 × 1580 D.

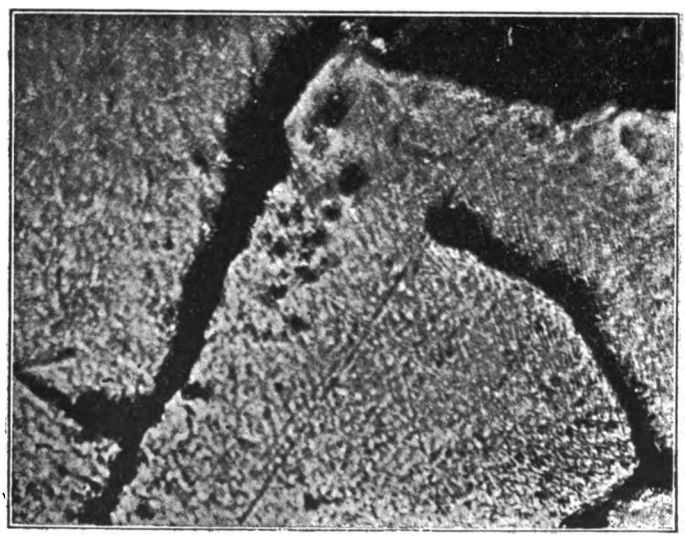

Fig. 4. More highly carburized iron, containing when fluid 7 per cent Carbon and 3 per cent Silicon. Combined Carbon 0.22 per cent. The section shows only ferrite und graphite.

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In conclusion, I wish to thank Sir William Roberts-Austen for the facilities he has placed at my disposal in connection with the experimental part of this paper, and Mr. E. O. Courtman, who prepared the sections and took the photographs.

THE MICROSCOPIC STUDY OF METALS.*

By W. M. STINE.

IN order to introduce this subject, which is somewhat recent in its development and not a matter of general knowledge, illustrations may be taken from the applications of the microscope to the work of the biologist and the physician.

The biologist employs the microscope in the examination of minute structural details. Not only the structural elements but their relations and groupings are the subjects of his investigations. In a restricted sense, a chemical analysis may afford some knowledge of the specimens; but this is not far-reaching and is rather a matter of secondary importance. One knows little of biological specimens from the knowledge of the chemical nature of their structural elements, the essential being rather the grouping of the structural elements. After the specimen is prepared for the microscope, differential chemical re-agents may be applied to enable the structural elements to be distinguished; or, recourse may be had to the simpler process of staining. Then, as a further means of investigation, heat may be applied or mechanical means may be resorted to that the physical relations of the structural elements may be changed.

While to a certain extent the microscope may be employed by the physician in an analogous manner, he usually employs it in a search for pathological, rather than biological phenomena. He wishes to ascertain what structural changes have taken place in the tissues of the body as the result of disease or injury.

These familiar illustrations will lead directly to another field of microscopical technology, in which the specimens are metals

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* *Journal of Applied Microscopy*, March 1900, page 786. The article is illustrated by two photomicrographs.

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and not organic tissues; and it may develop that these apparently dissimilar subjects have a very great deal in common, so far as the methods and even the ends sought are concerned. Until recently the applications of the metals to the arts and sciences, have been based almost wholly on the results of chemical analyses and certain physical tests involving, for instance, the limits of elasticity, and ultimate breaking strength.

Great efforts have been made to establish the physical properties of steel, for example, as a function of its chemical constitution, but, as one might readily infer, the success attending this has been only partial. Without desiring to be tedious, it may be well at this point to consider some of the demands the metallurgist is compelled to meet at the present time. It will no longer answer to use steel in framing a structure, and then more steel to make certain of it; efficiency requires that not a ton of metal above the amount actually required shall be used. Such conditions demand a complete knowledge of steels and other metals for structures, while the requirements of machinery are no less exacting.

In order to reduce friction in machinery, soft bearing metals are widely employed, which are usually an alloy of two or more soft metals such as tin and lead. This matter of friction is of such vital importance that it has become necessary to obtain a more complete knowledge of bearing materials than can be gained from their chemical analyses.

Chemical analysis can not take account of the physical treatment of metals in the processes of their manufacture. Drawing, rolling, hammering, and annealing, and other causes induce changes of structure which profoundly affect the elasticity and other physical properties, though they do not in the least change their chemical constitution. The results of impact and excessive strain can only be thoroughly known when the changes of structure which these causes have induced in the metals affected, are fully known. The investigation of such details corresponds closely with the search of the physician for pathological changes. The recent application of the microscope to the study of metals has proven so advantageous that it is probable that the microscope will become as necessary an instrument for those who apply the metals structurally or mechanically in an exact and scientific manner, as it now is for the practice of the physician.

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There is frequently to be found in different specimens of wrought iron a failure to develop the degree of strength and elastic qualities which are to be expected from a metal of its chemical purity. By filing off a surface with the grain of the iron and etching it with HCl, the gross fibrous structure becomes apparent and layers of slag are to be made out; yet no precise information is to be gained from such a method. A more painstaking preparation of the surface, with greater care in the etching, will yield a specimen which may be subjected to a high power objective. Under its resolving power the crystalline structure can be seen and the exact manner in which the fine threads of slag interfere with the homogeneous fibre of the iron. The metallurgist in this way is enabled to see the faults in his material and even to make quantitative estimates of the constituents presented to his view. In a general way this process may be applied to all metals, and places those who work with such materials in the possession of an exact knowledge of their intimate structure.

The influence of working on soft iron is easily traced by the aid of the microscope. When iron is compressed by traction through a steel drawplate, the original fibrous structure due to polygonal crystals is changed, especially on the outer layers, by the crystals becoming lenticular, as is readily seen under the microscope. If such iron is annealed its original ductility is usually restored; and it is an interesting question to determine by an examination, the extent to which the changes in the physical properties are functions of the changes in the structure. Specimens of soft steel which have shown the lenticular form of crystals after compression, upon annealing for two hours at 650° C. have been shown to have lost the lenticular shape of the granules; the crystals then being more or less equi-axial, resemble in this condition the crystals of ordinary iron.

Probably the more important results have been obtained from the study of alloys for their physical constitution; and the connection between this and their physical properties has always been involved in more or less obscurity in spite of a vast amount of investigation. The alloy of pre-eminent importance at present is steel, the variety of which both in structure and properties has been bewilderingly large. It is true that much has been learned about steel in the past, but the knowledge has been largely

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obtained through empirical methods. Chemical analysis has not been without great value, but wide physical variations in steel have been found to exist where there was practically no chemical variation which the processes of analysis at least were able to detect. With the development of the processes and methods of metallography, the metallurgist began for the first time to acquire a somewhat exact knowledge of the steels; not only their true composition, but their physical properties as functions of their structure and composition. The technology of the subject of the metallography of steels is so extensive that it cannot be entered upon even in brief detail in such a limited article as the present one. Without enlarging upon the possible development of the metallurgy and physics of steels under the impetus of metallographic studies, it can yet be foreseen that we must eventually realize from them all the immense advantages that accrue from an exact knowledge of the structure and composition of these alloys and their physical properties as functions of these details.

Roberts-Austen more particularly has devoted himself to the study of the physics of the alloys of two and three metals, and his investigations are familiar to most physicists, and their value is generally appreciated. Later, Stead and Charpy* have added to the knowledge of alloys by their studies of the industrial alloys. From their work a bearing metal composed of tin, lead, and bismuth upon an examination of a section shows: 1. large areas of metallic bismuth with indications of grains of bismuth which have solidified between 175° and 125° C.; 2. around each grain of bismuth there existed a zone in which particles of bismuth alternated with particles of tin; 3. and this structure was held in a matrix of the eutectic mixture of the three metals.

The function of the bearing metals as a means for reducing friction and the wear of journals is very clearly defined from the micrographic study of their sections. The load of the journal is carried through them by the hard grains of the alloy, which have a low co-efficient of friction, the plasticity of the matrix making it possible for the bearing to adjust itself closely around the shaft.

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* G. Charpy in *Bulletin Société d'Encouragement pour l'Industrie Nationale,* June 1898. — *The Metallographist,* Vol. II, page 9.

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From this general survey it must be apparent, to those not versed in the technology of metallurgy, that an adequate foundation exists upon which a new branch of science may be established, which must become of great value both to the pure physics of the structural constitution of metals and alloys, and especially to their applied physics. The new science rejoices in the name Metallography, a well-established term, which only questionably connects the numerous phases of the things it is supposed to designate. The development of the science is involved with the selection of the name for its designation. As formerly applied, metallography referred to a species of lithography in which etched metal plates replaced the stones of the other printing process. The earliest attempts at a study of the structure of metals were made by etching plates of iron or steel and obtaining some impression on paper from them. This process yielded merely a description of the gross anatomy of the specimens studied, and was of limited value. Presently, by an extension of the process of etching, specimens were obtained so finely grained they yielded only to the resolution of the high-power objective. Then metallographs became instead, micrographs. The recognition and extension of the micrographic process was the real beginning of the new science of the minute structure of metals, and has within the past ten years made wonderful progress. It is true that micrographs of metals had been made for many years prior to this recent advance, but no concerted investigation resulted. As the term metallography is now used, it denotes an increasing mass of knowledge obtained rather by the use of the microscope than methods dealing only with the gross anatomy of the metals. The practician of the new science is termed a metallographist.

The Preparation of the Specimen. — As in biological technology, the specimen must be cut down until the section desired for examination is reached. The area of the section is usually small, not exceeding one square centimeter.

The specimen bit of metal is first cut away by filing, sawing, a machine tool, or by grinding against an emery wheel, and it is then rubbed on emery cloth and paper of increasing degrees of fineness. Ultimately it is polished on leather with fine jeweler's rouge, or it may be buffed against a leather polishing wheel.

The specimens of the harder metals present few difficulties

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in their preparation and their surfaces polish readily. But many alloys are difficult to manipulate, especially when they contain lead or tin; these soft constituents are apt to spread or run over the surface of the specimen, making a film which will neither take a specular polish itself nor permit the process of polishing to extend to the harder constituents underneath. Emery paper can not be used on such metals, but their surfaces should rather be scraped plane.

If the constituents in the alloy are differently colored, polishing alone will be a sufficient process to enable them to be distinguished, but this is rarely the case; much less can the intimate structure of the harder metals be brought out by examining their polished surfaces. The microstructure is only developed by some etching treatment after a preliminary polishing. If the specimen is to be subjected to mechanical or thermal treatment in order to observe the structural changes thus brought about, the identical etching process must be employed throughout.

A specimen not to be subjected to further tests may be treated with differentiating re-agents and its various structural elements may in this manner be readily distinguished. The etching re-agents commonly employed are acids, alkalies, and alkaline sulphides, when possible applied in dilute alcoholic solutions in order to remove grease left from the polishing process. That the etching may proceed uniformly over the entire surface it is necessary that this should be scrupulously cleaned. The cleansing calls for the exercise of considerable care and judgment, avoiding the use of any cleansing re-agent which will produce the slightest etching effect. The specimen, too, after cleansing, must not be dried at a temperature which will cause oxidation of any structural constituent. It is scarcely necessary to add that the surface should not be touched with the finger at any stage in the cleansing or etching.

Electrolysis in a saline solution affords an additional and a rapid method of etching and is generally used, the specimen, of course, being connected with the positive pole of the battery. In case any of the constituents are readily oxidizable and show characteristically colored films under the treatment, the specimen may be slowly heated until the desired tint is obtained. This treatment is found to be useful in alloys containing antimony, lead, or copper. In the same line, differential coloring is often a

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ready means for the detection of certain constituents; tincture of iodine or any similarly acting re-agent may be employed, the method being one which is capable of considerable extension and variation.

No especial form of microscope is needed for the work; any stand may be used which has preferably a large opening in the ocular axis of the stage. The stage, in short, should be of such design that specimens, often fragments of metal polished and etched on one side and of irregular shape, may be held in proper position for examination.

The lighting is a difficult feature and may be accomplished by the use of totally reflecting prisms, with or without condensing lenses. Doubtless the demands of this new branch will call out, from time to time, appropriate modifications in the microscopic stand and special auxiliary apparatus and attachments.

The new science has already in this country an excellent journal devoted specifically to its development, which was established in 1898. Its files will afford an accurate survey of what has already been accomplished and will give all the needed instruction for these interesting tests. It is called *The Metallographist,* and is edited by Albert Sauveur, who has been one of its foremost investigators.

On Mysterious Fractures of Steel Shafts.*— Mr. R. Schanzer says that the phenomenon of sudden and unexpected fractures in steel, and particularly in shafts and other steel structures which have been subjected for a considerable length of time to shocks and vibratory stresses, is, no doubt, of a most perplexing character, and has, therefore, attracted the attention of many among the most authoritative investigators. Nevertheless, the question is as yet very far from being solved, and many sides of the problem are still lacking satisfactory explanation.

In fact, the mysterious fractures alluded to can be said, after all, to represent nothing else than so many cases of more, or less, marked brittleness, i.e., of a phenomenon which our present knowledge is quite inadequate to account for, except in a very limited number of cases.

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* Read before the Institution of Naval Architects, April 6, 1900.— The paper was illustrated with a number of photomicrographs.

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Moreover, another very startling feature of the phenomenon ought to be noticed: very frequently sudden fractures are produced in steel shafts, as well as in other engineering structures of steel which have been accurately tested before use, the ordinary physical tests having uniformly pointed to the best quality of metal; after such a structure has been working continuously for several years in the most satisfactory manner, fracture suddenly occurs; no gradual, visible deterioration having afforded any indication of coming danger up to the very moment of the failure.

In this latter respect the question is very closely related to that of fatigue, which forms, no doubt, one of the most important, and, at the same time, one of the most disputed problems to be met with in the study of the resisting properties of metals. In fact, notwithstanding the very valuable work of Wöhler, Sir Benjamin Baker, Professor W. C. Unwin, and many others, and the recent and extensive researches of Mr. Thomas Andrews on the deterioration of metals by fatigue, the matter can by no means be considered as settled.

To quote but a single instance, it is still a disputed question whether vibratory stresses far below the elastic limit of a given metal may be capable of modifying its microscopic structure; however, for the purpose of the present paper it is not quite necessary to answer such a question.

There is one fact which cannot be doubted in any case, and that is the unquestionable influence of the pre-existing structure, which may be either favorable, or opposed to the effects of that peculiarly dangerous action which is generally known as *fatigue*.

Moreover, in the author's opinion the term fatigue is very frequently considered as an easy way to overcome the difficulty of accounting for so-called mysterious fractures. In reality, all known causes of brittleness are favorable to the deterioration of fatigue. Among such causes the following may be quoted: — Abnormal crystallization, inducing internal stresses by which the metal is rendered brittle; immoderate size of crystals or grains; sharp junction lines or insufficient cohesion between crystal-faces; layers of various impurities arranged in the intercrystalline spaces; microscopic flaws due to different causes, etc.

The purpose of the author is to describe one more of such causes, viz., a particular brittle structure which he observed in

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a suddenly fractured shaft that was presented for his examination. To tell the truth, a similar structure has already been mentioned in a paper read before this Institution by one of its most eminent members, Mr. A. E. Seaton,* which contains a very valuable report by Professor J. O. Arnold.

However, in the shaft now mentioned, the particular structure alluded to was to be noted under a quite characteristic aspect, differing in some respects from that described by Professor Arnold. At any rate the author could not help remarking a rather startling analogy between the two structures.

Before stating the results of his own observations, Mr. Schanzer gives a short abstract of the report of Professor Arnold.

The shaft forming the object of the present paper was supplied about eight years ago by a first-class French firm, and worked satisfactorily for the whole time, till it was removed (some months ago), owing to a deep crack which had suddenly appeared on its surface.

Moreover, the whole surface of one of the necks was covered by very fine hair cracks running very close to each other, parallel to the axis of the shaft; these cracks were very clearly seen on the surface of the shaft.

Microscopic Examination. — Samples were first taken from the broken shaft without any preconceived plan, and with the sole purpose of getting a general idea of the structure of the metal.

A segment was detached from the fractured surface; the segment was divided into pieces by five parallel cuts; finally five of the pieces obtained were polished on the plane surfaces generated by the saw. These happened to be parallel to the axis of the shaft.

Under the microscope the five sections showed a very characteristic structure.

The first thing noted was the pronounced separation of constituents, and it was just this marked separation of ferrite from pearlyte which recalled to the author's mind Professor Arnold's report, and, considering that the samples referred to by Professor Arnold came from a shaft whose history, in many points, re-

* "The Causes of mysterious Fractures in the Steel used by Marine Engineers as revealed by the Microscope," by A. E. Seaton. — *Trans. Institution of Naval Architects,* Vol. XXXVII, page 210.

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sembled that of the shaft which he was examining, he could not but find a very startling analogy between the two cases.

On the other hand a very important difference was to be noted at once. In the micrographs of Professor Arnold the structure was formed of patches of pearlyte, irregularly distributed upon a ground consisting of ferrite, while in this case the appearance of the structure was quite different. Separation of constituents was to be noted here as well as in the instance dealt with by Professor Arnold, but it was effected in a different way. The structure could be said to be formed of dark and bright stripes alternately, and very elongated, in a given direction.

The dark stripes were readily seen to be formed by a ground of ferrite on which small areas of pearlyte were very densely distributed; the bright stripes were pure ferrite.

In both of the structures referred to (viz., that dealt with by Professor Arnold, and that observed by the author) separation of constituents is noted, shown on a micro-section by areas of alternately hard and soft metal: this condition, forming the common feature of both instances is, as already remarked, very favorable to brittleness. But two important differences are to be noted between the two structures: the first relates to the constitution of the hard areas, the second regards their shape. The soft areas are formed in both cases by ferrite; but the hard areas are formed of pure pearlyte in the shaft examined by Professor Arnold and of pearlyte mixed with ferrite in the other instance.

In other words, the hard areas are harder in the case referred to by Professor Arnold, than in that examined by the author. This first difference is very simply accounted for by the different carbon percentage, which is much higher in the shaft examined by Professor Arnold.

The second difference relates to the shape of the areas. The stripes of alternately hard and soft metal were all elongated in one and the same direction, which is by no means the case with the micrographs obtained by Professor Arnold, where pearlyte forms irregular patches spread out upon a ground formed of ferrite.

It ought, however, to be stated that the micrographs of Professor Arnold represented sections normal to the axis of the shaft, whilst the author's section was parallel to the axis. Such a fact, Mr. Schanzer thinks, can fairly well account for the difference

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observed. Indeed, supposing that, in the shaft examined by Professor Arnold pearlyte was to be found in the shape of solids elongated in the direction of the axis of the shaft, a normal section ought to show the appearance seen in the micrographs of Professor Arnold, whilst a section parallel to the axis would have shown the hard areas (pure pearlyte in that particular instance) in the shape of elongated stripes, precisely as is the case with the shaft examined by the author.

On the other hand, if this view of the matter be correct, it would be expected that sections from the author's shaft, taken in the same way as those of Professor Arnold, i.e., normally to the axis, would not show the stripes noticed.

The results of microscopic examination proved conclusively that matters lay just as anticipated by the author.

Twenty-four samples were polished parallel to the axis of the shaft and showed a quite noticeable similarity of structure.

On the other hand, as was anticipated, in twelve samples polished normally to the axis of the shaft the parallel stripes were no longer to be seen. There were bright areas formed of pure ferrite, dark areas formed of pearlyte mixed with ferrite, exactly as in the twenty-four samples previously alluded to; but neither the dark nor the bright areas assume the shape of elongated stripes.

This structure is analogous to that of the micrographs of Professor Arnold, the difference between the two structures consisting, as already said, in the fact that, in the latter steel, which was considerably harder, the irregular patches were formed by the hard metal (pearlyte), while the ground mass consists of the soft one (ferrite). The reverse phenomenon took place in the shaft under examination, where the irregular patches are formed by the soft metal (ferrite), whilst the ground mass is formed by the hard one (a mixture of ferrite and pearlyte).

Knowing both the longitudinal and the transverse section of the structure, we are obliged to conclude that the metal of the shaft under examination was formed of solids consisting of ferrite, elongated in the direction of the axis of the shaft and imbedded in a harder mass, being a mixture of ferrite and pearlyte.

Chemical Analysis. — It was desired to ascertain whether any of the causes which, in the instance described by Professor

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Arnold, had contributed to the failure, were to be met with in the present instance, in addition to the unfavorable arrangement of constituents which we already know to be a feature common to both structures.

It was, therefore, wanted in the first place to know whether there had been liquation; with this object a series of samples for chemical analysis were taken, both at the centre and near the circumference, in order to ascertain whether the chemical composition was uniform throughout the mass. A series of samples on the whole length of a radius of the fractured surface was also taken.

Microscopic examination had shown the utmost uniformity of structure throughout the whole mass of the shaft, and chemical analysis proved that also the chemical composition was of the greatest desirable uniformity.

Therefore, one of the possible causes of failure which were found in the case of the shaft examined by Professor Arnold, viz., liquation, was totally absent in the present instance.

If the other causes mentioned by Professor Arnold as having possibly contributed to the failure of the shaft forming the object of his report, are considered, it is found that the carbon percentage of the shaft under examination was very low, varying from 0.155 to 0.170, i.e., far lower than that found in the shaft examined by Professor Arnold (from 0.310 to 0.470 per cent), and even lower than had been specified for the latter shaft (0.20 to 0.25 per cent). As to the sulphur (varying from 0.055 to 0.150 per cent in the shaft referred to by Professor Arnold), in the present instance it was met with in a very satisfactory proportion (minimum, 0.023; maximum, 0.038; mean amount, 0.027 per cent). The phosphorus was rather high in both metals. Nevertheless, the high percentage of this element which was found at the periphery of the shaft dealt with by Professor Arnold was never reached in the other shaft, which shows a mean phosphorus percentage of 0.092 per cent, the minimum being 0.082, and the maximum 0.101.

Conclusions. — From the above comparison of the properties of the two shafts referred to, it is to be clearly seen that there are but two features common to both metals, viz.: —

1. The unfavorable arrangement of constituents.
2. High phosphorus percentage (though in the shaft ex-

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amined by the author the said element was far from reaching the high amounts met with in the shaft referred to by Professor Arnold).

The reasons why the unfavorable arrangement of constituents, just mentioned, is liable to induce marked brittleness in steel were very clearly explained by Professor Arnold in the paper referred to.

Besides, the author thinks, it ought to be clear to anybody that sharp junction lines between two metals of very different hardness (which was precisely the case both in the shaft examined by him and in that referred to by Professor Arnold) must necessarily cause a tendency of the two metals to slip upon each other under vibratory stress. In other words, the surfaces separating the said two substances of very different hardness are certainly surfaces of weakness.

If it be supposed that the masses of different hardness, of which the metal was seen to be formed, assume the shape of solids approximately rectilinear, and very much elongated in a given direction, such circumstance can but greatly increase the brittleness.

Moreover, in this case the intersections of the surfaces of weakness just mentioned, with the external surface of the shaft will, approximately, coincide with the generating lines of the latter, thus giving rise to cracks parallel to the axis of the shaft. Now, the existence of the said cracks as well as the stripes of alternately hard and soft metal was proved by the examination of the shaft. Moreover, there are strong reasons for supposing that the alternate stripes were present also in the shaft examined by Professor Arnold (the hard metal being formed of pure pearlyte in the latter case). Only they did not appear, as the sections examined were all taken normally to the axis of the shaft.

As regards the cracks alluded to, they were observed by the author, not only on the shaft dealt with, but on two more rolling mill shafts likewise broken by fatigue. In both cases the cracks were parallel to the axis. Also, Mr. A. E. Seaton alludes to a broken shaft showing hair cracks on the surface. It would be interesting to know whether the direction of the cracks was parallel to the axis also in this instance. That would be a confirmation of the preceding statements.

Supposing the views set forth above are correct, another

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question arises, which is of the utmost practical importance: "What are the reasons for the particular arrangement of constituents described?" Unfortunately, the author fears that an answer to this question cannot be easily obtained at the present stage of our knowledge. It could, perhaps, be thought obvious to inquire whether the cause of the particular structure observed should not be sought for in the action of fatigue. That would imply that the vibratory stresses to which the shaft was subjected, when working, might probably have caused a re-arrangement of the microscopic constituents of the steel. That, however, would be quite impossible, as the definite arrangement of ferrite and pearlyte is completed at Brinell's point V, far above any temperature attainable by the metal when working, even if strongly heated by friction.

The author is consequently led to conclude that the stratified structure, observed in the shaft referred to, could by no means be originated by fatigue, though it certainly was favorable to the development of deterioration of fatigue under the prolonged action of vibratory stresses.

With regard to the second disease noticed in the shaft, the high phosphorus percentage, it might, possibly, be advanced that in both shafts phosphorus brittleness was the chief cause of the fracture. Phosphorus brittleness may certainly have played a part even with the shaft forming the object of this paper, the phosphorus contents of the latter being unquestionably excessive, though considerably lower than in the shaft examined by Professor Arnold. But, on the other hand, the deleterious mechanical effect of a structure, such as has been described above, can by no means be overlooked, and is moreover proved by the presence of the hair cracks largely dealt with in this paper.

The author, therefore, thinks that the importance of the stratified structure, as a cause having induced brittleness in the shaft referred to, is by no means impaired by the fact that another cause of brittleness — viz., high phosphorus — was present in the same metal. It must, however, be recognized that the simultaneous presence of high phosphorus and of the stratified structure is, perhaps, not altogether fortuitous.

To sum up the experimental evidence afforded by the examination of the two shafts and the brittle plate dealt with, the author thinks that the three instances mentioned concordantly

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point to the harmfulness of the stratified structure. As regards the influence of phosphorus upon the formation of the said structure, there is nothing impossible in such an assumption, though it is very far from being proved by the few instances quoted. At any rate, it is quite clear that the influence of phosphorus would only account for the marked separation of pearlyte from ferrite, whilst the arrangement of the separated constituents in parallel stripes would be, of course, attributable to the direction of forging, in the case of shafts, or of rolling in that of plates.

The author briefly recapitulates the results of his research as follows:

First. The particular structure dealt with in the present paper was observed by the author upon forty-one samples of a steel shaft suddenly broken. Such structure is always characterized by a marked separation of ferrite and pearlyte, the latter gathering, preferably, in given points of the section, so that areas of different hardness are formed.

Moreover in the case of samples polished parallel to the axis of the shaft, the said areas take the shape of elongated stripes, parallel to the direction last mentioned. The latter structure gives rise to *hair cracks* running in the same direction on the external surface of the shaft.

Secondly. In the shaft dealt with by Professor Arnold, separation of constituents is likewise observed. The hard areas are formed of pure pearlyte.

The stratified structure could, however, not be detected in the shaft examined by Professor Arnold, no samples polished parallel to the axis having been taken from the same.

Thirdly. The stratified structure referred to was found by me to be quite distinctly developed in a series of samples taken from a very brittle steel plate.

Fourthly. The parallel hair cracks indicative of the stratified structure, alluded to, were met with, not only in the shaft already referred to, but in two other rolling mill shafts which were both broken in a sudden and unexplained manner. In both of the latter shafts the direction of the cracks was found to be parallel to the axis, exactly as in the case of the shaft previously referred to.

Fifthly. The presence of hair cracks is likewise mentioned in the case of the two shafts alluded to by Mr. A. E. Seaton,

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one of which is that examined by Professor Arnold, but he does not state whether such cracks were, or were not, parallel to the axis of the respective shafts.

Sixthly. Nothing can be said as to whether high amounts of phosphorus are favorable to the development of the particular defective structure described in this paper. Nor can any other cause for the production of such a structure be suggested.

Of course, by the preceding statements I do not mean to assert that the structure described in this paper must necessarily be found in every suddenly fractured shaft, for the possible causes of deterioration by fatigue are of an exceedingly manifold nature. It is, however, quite possible that the structure alluded to should be met with not only in the few instances quoted. The fact that such a structure has, up to now, escaped the attention of observers is very easily accounted for. First of all micrographic examination of defective structures is, as yet, very far from being practised in all such cases where it would be desirable to do so. Besides, the most characteristic feature of the phenomenon, i.e., the stratified structure is noted only on samples polished parallel to the axis of the shaft.

Perhaps if the method of examination described in this paper were adopted in other instances of suddenly fractured shafts a similar structure would be met with in some cases.

A similar method of examination ought to be also applied to other steel structures, as plates, rails, etc. Of course, in the latter case the direction in which the samples are to be polished must be modified according to circumstances.

This line of research would perhaps prove useful, in order to get further information upon the subject dealt with in this paper. I am quite aware that experimental evidence is needed before the conclusions set forth may be considered as definitive. I, however, am satisfied with having called attention to a series of observations in order to induce others to direct their researches to the same subject, and in the hope that such researches will supply new evidence in support of the hypotheses I ventured to advance in the course of the present paper.

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The Physics of Steel.* — The century opened with the expression of fundamental but comparatively simple views as to the constitution of steel. The importance of carburization had been accepted and the protean nature of iron itself was recognized by Bergman's classical phrase, "*polymorphum ferrum.*" As the century closes it is seen that iron has gathered to itself, often in considerable volume, numerous associates with which it was originally entrusted only in small quantities. Industry has consequently been enriched by varieties of metal possessing properties hitherto unknown. These properties have been investigated by a host of workers, and the mention of only a few names may seem to be invidious. Nevertheless, I am satisfied that when writers come to look back to the end of this century, as we do to its beginning, the phenomena connected with iron and steel to which they will revert will be comprised in the words "points of transformation." Our country will receive its share of these retrospective thoughts, for the names of Gore and Barrett are waymarks. Tchernoff will be remembered, for he proposed a thermometric scale by which manufacture of steel could be controlled.

From among Frenchmen one name will arise in the memories of future historians of metallurgy, and that name will be Osmond. It will not be forgotten how much Osmond did in defining the modes of existence of carbon in steel. It will be remembered that by his observations a single point of recalescence became resolved into many points. He traced the influence of hysteresis and showed how the position of the points of transformation vary within a certain range of temperature with the element with which the iron is allied. He showed that if a certain variety of steel has been quenched at a point above or below its critical point, it may present two states of equilibrium. These may be widely different, either as a consequence of allotropic change in one of the constituents or in the chemical grouping at the moment the metal passes the critical point. I am not unmindful that M. Grignon, who translated Bergman's "De Analysi Ferri" into French, shared his views as to the allotropy of iron, nor do I forget that Cizancourt pronounced advanced views on the subject in 1865, as did also Tait in the Rede Lecture delivered at Cam-

* Abstract from Sir W. Roberts-Austen's presidential address before the Iron and Steel Institute, Paris Meeting, September 1900.

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bridge in 1873. Osmond, in fact, insisted on the allotropy of iron, proved its fundamental importance, and identified himself with its study, but that, though it is his great achievement, is far from being his only one. He showed that the properties of steel are a function of the cycle of temperatures to which it has been subjected. There have been many workers whose investigations possess strong individuality and significance peculiarly their own whose results nevertheless intensify the interest of Osmond's labors and conclusions. I hope I may be forgiven the brevity with which I cite these. Pionchon showed that at a temperature of 700° the specific heat of iron is altogether exceptional. M. Guillaume studied a series of nickel steels in which the transformations are reversible. This reversibility is associated with some important peculiarities as regards expansibility. Henri Le Chatelier pointed to magnetic irreversibility in the case of certain manganese steels. He moreover examined the influence of the gaseous atmosphere surrounding the steel in which the points of transformation occur. Charpy studied the hardening of steel with reference to the critical points. Tomlinson showed that the molecular change in iron is revealed by a change in magnetic properties. Madame Curie showed that the allotropic change effects a true magnetic transformation. Dumont recently pointed out that in nickel steel the point at which magnetic transformation occurs depends on the proportion of nickel present, while experiments by Guillaume and by Dumont indicate that chromium acts like carbon on certain irreversible varieties of steel and lowers the temperature at which magnetic transformation occurs. Carnot and Goutal isolate various compounds which in special varieties of steel give rise to variations of properties. The great importance of critical points in relation to the dimension of grain in steel, and consequently on its strength and extensibility, have been shown by Brinell, by Sauveur, and by Morse.

The second group into which modern investigation may be divided involves the use of the microscope, and the application of this instrument to the metallurgy of iron is less recent than it is often supposed to be. Réaumur in 1722 describes the structure of a chilled casting under the microscope, and he traces the changes in the structure of softened cast iron as modified by the elimination of impurities. François, again, so early as 1833,

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took the very interesting case of the direct reduction of iron from its ores, and followed the successive changes by the aid of the microscope. The following passage, translated from this quaint and accurate observer, is full of interest, and might almost have been written by any of us to-day: —

"If to these analytical data observations under the microscope with a magnification of 300 to 400 diameters be added, it is seen that ordinary iron is merely a metallic network with a close-grained tissue with submerged scoriaceous opaline, sometimes subcrystalline, portions, and with little globules and metallic grains ranged in every direction. Sometimes nests of translucent prismatic and bacillary crystals, with metallic portions adhering, are noticed hidden in the paste. These are the grains of steel which can be made to disappear by heating."

England in turn in 1864 gave a new impetus to this important branch of investigation by the labors of Sorby, in whose admirable work polished and etched sections were employed. The microscope as an implement of research hardly received due recognition until Osmond published a classical series of researches which have earned the lasting gratitude of investigators. In no branch of my subject do I so much regret that the limitations of space have obliged me to confine myself to the bounds of our two countries, as I should like to have dwelt on the micrographic work of Martens, Sauveur, Behrens, and many others. I will only add that the work of Stead deserves special recognition, not only for its minute accuracy, but for its originality. Quite recently Ewing and Rosenhain have obtained most interesting results, which go far to explain by micrographic evidence the elasticity and flow of metals, and also the mode by which crystals grow in metallic masses.

A Report of the Departmental Committee on Steel Rails recently issued is the first Government publication in England in which the importance of micrography has been recognized.

Scientific discoveries have during the century worked a complete change in our intellectual appreciation of natural phenomena, but in our work the standpoint of the new century will be singularly like that of the old. A few tenths per cent of carbon hidden in iron still dominates our branch of metallurgy. Bergman recognized this before the nineteenth century began, and insisted both on the polymorphy of iron and the importance of

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carbon in relation to it. He pointed out, moreover, that in every great change effected in the constitution of iron, heat is either absorbed or evolved. The sentence in which Bergman sums up the results of his own calorimetric experiments is full of interest: — "Si præcendentis momenti allata experimenta considerantur, haud difficulter elucet, quemlibet statum respectu caloris absconditi circumscriptum esse."

Allotropy and carburization of iron will be the first passwords of the twentieth century, and Osmond, the great allotropist of the age, has blended their significance by showing that the power iron has to retain carbon in solid solution depends on the particular allotropic form in which the iron exists. There is no other fact which so profoundly affects the industry of the world.

We now know, but only at the end of the century, that in steel of somewhat low carburization, but wide industrial application, that the heat equivalent of the allotropic change is the double of that due to the change of the relation between the carbon and the iron, being 18 and 9 calories respectively.

As one of the last important engineering works in a century which the Eiffel Tower would alone have made remarkable, I may refer to a great work in which a different material is used from that which is employed in this great structure. The Alexander III. Bridge, which crosses the Seine in a single span, is of cast steel, and 2200 tons have been employed in its construction. If the steel used in such a structure is heated to a temperature of 1000° and is "quenched" by rapid cooling in air to about 600°, the metal so treated becomes stronger, more elastic, and resists shock better than if it had been annealed. Hence the material adopted in the great work which so intimately expresses the genius of the Exhibition of 1900 affords the last cognate illustration of the importance and accuracy of Osmond's views.

I must not omit some reference, though it must necessarily be brief, to the metallurgical literature of our two countries. This has been greatly enriched by the admirable treatise on "Metallurgy" written by Dr. John Percy, which developed into a series of volumes, containing no less than 3500 octavo pages, the last of the series appearing in 1880. He was, it will be remembered, President of this Institute during the years 1885-87. His volumes are remarkable throughout for minute accuracy, and for the care with which the illustrations were prepared, al-

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most every woodcut being a trustworthy though small measurable drawing. He has given us, in terse and vigorous English, a priceless record of many early processes, and ones which, though later, are obsolete. He was firmly impressed with the fact that metallurgical problems demand for their investigation the exercise of the highest analytical skill, and involve considerations worthy of those who delight in transcendental inquiries. He effectively quotes Réaumur's remark, "L'utile bien considéré a toujours du curieux, et il est rare que le curieux bien suivi ne mène pas à l'utile." I have elsewhere observed that he studied in Paris, making the acquaintance of the leading French chemists of the time, who doubtless directed him much towards the line of work to which his life was mainly devoted. Percy may fairly be said to have originated the English literature of metallurgy, for until his time the works on metallurgy were slender and far between. This was not the case in France, where the works of Rivot, the admirable and profusely illustrated treatises of Jordan, as well as those of the great chemists I have named earlier in this address, were well known. France, moreover, possessed in the *Annales des Mines* a veritable storehouse of knowledge, which has done for the iron and steel industries throughout the century what our own Journal has attempted to do in the latter half of it. I would only add, that if Percy has been the historian of metallurgy, Gruner systematized it. I would gratefully offer my testimony to the value of his "Traité de Métallurgie" as a model of scientific exposition and methodical criticism, which enabled the tangled threads of metallurgical practice to be unravelled. Few men have more effectively combated than he did the assumption of prejudice and mystery which all branches of the metallurgic art too long retained.

There are many subjects you will regret that I have not touched upon. The question of naval defensive armor, for instance, might well have been referred to even on so eminently pacific an occasion as the last Exhibition of the century, but I will content myself with reminding you that France was the pioneer in the employment of naval armor, and that the launching of the armor-protected *La Gloire* in 1860 gave the signal for the reconstruction of European navies.

The extraordinary development of the use of iron for constructive purposes revealed by the present Exhibition, demands

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that some reference should be made to the relations of iron and steel in art, with which industry and science are so closely connected.

> " And trade is art, and art philosophy,
> In Paris,"

as Mrs. Browning reminded us. The vast array of ephemeral buildings which line the Seine has been compared collectively to a huge organism, the bones and fibres of which are of iron and steel, covered with an investing body of plaster that has served as a basis for adornment of all kinds. There are some who doubt whether, from an architectural point of view, the lavish use of the new resources which metallurgists have afforded has been successful. A subtle critic, M. Robert de la Sizeranne, in a brilliant paper recently published, entitled, " L'Esthétique du Fer," takes a despondent view of the result, and considers this new use of iron to be " à la fois le triomphe du progrès scientifique et son châtiment." But when he turns to the beautiful bridge, which, as I have already said, is an exponent of the genius of the Exhibition, he writes enthusiastic words, which I may well quote, and are as follows: — " Nous ne quitterons pas les bords du fleuve sans avoir senti la vie. Car elle est dans ces formes admirables du pont Alexander III. Ceci aussi c'est de l'art."

For my own part, being a metallurgist, I cannot expect that any importance will be attached to my individual opinion on an æsthetic question, but I would venture to observe that ideas of beauty are instinctive, and that pleasure in the contemplation of any work, whether of nature or art, depends on delicate and untraceable perceptions of fitness, propriety, and relation. This being admitted, I find myself unable to stand under the Eiffel Tower, and follow the course of the curved lines, which suggest the details of an immense but delicate coral, without being carried away by the impression that the structure is not only wonderful but beautiful.

The part played in the industrial evolution of the world by iron and steel metallurgists will be evident to all. The fact that they have contributed with commensurate success to the advancement of pure science is less generally known, and I may well, therefore, devote a few words in conclusion to recording some instances which readily present themselves. The carburization of iron offers the first case in which the diffusion of

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solids in solids was observed. The study conducted by metallurgists of the associations of carbon and iron affords the most complicated case yet known, and indeed the only one which has been worked out, of metallic solid solutions. It presents, moreover, a unique case of the importance of allotropy in connection with metals. In 1000 parts of steel 997 parts, more or less, may be iron and the rest carbon, but the result of the union is a metal that is more widely used and has more varied attributes than any other. It would seem that nature has enshrined in steel a series of her most complex secrets which we must discover as a condition of our successful use of it. Metallurgists have carefully investigated, and conduct daily, operations of vast industrial importance, in which the presence of a third body enables either elementary bodies or compounds to react on each other. Habitually conducting operations at high temperatures, which often invert ordinary chemical reactions, has led to the acquisition of a mass of information as to the reactions that do occur, and the compounds that are formed under such conditions. Pyrometric records have, moreover, enabled the equilibrium of the less fusible metals in their liquid as well as in their solid state to be studied — a branch of work which chemists generally have not been in a position to undertake. The necessity for submitting the physical and mechanical properties of iron and its alloys to rigorous tests, as a routine operation in works, has afforded a rich store of information as to the molecular constitution, not only of metals, but of matter generally. Metallurgists have to deal with cases in which a mass of metal is acted upon by added matter in proportions that are too minute to intervene directly by the formation of chemical compounds with the whole of the mass, while in some cases no compounds are formed. The influence of the atom must, therefore, be more or less directly exerted. Hence it is that we, who still conduct processes, the traditions of which came down to us from mythological times, have done our share in "giving the old Greek atomism a modern and a higher consecration."

We hold this meeting as the representatives of the greatest industry the world has seen. We are more than this, for we are the exponents of the scientific and industrial union which subsists between our nations, and of national appreciation that has remained unabated though tumults have shaken our peoples. May both nations in the coming century not forget how much we

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owe to the old Greek guidance. The Greeks gave us the name Siderurgy, by which our art is known in France. Plato, moreover, told us that the god who moved men through their sympathies inspired the invention of all industrial art, "the melody of the Muses, the metallurgy of Hephæstus, the weaving of Athene"; and if you have followed this brief address you will realize how much metallurgical progress has owed to sympathy — the great interpreter which joins the industrial workers of France and England in a bond of peace that is stronger than iron, truer than steel, and yet is one which is sufficiently extensible to embrace all our peoples.

CORRESPONDENCE.

To the Editor of *The Metallographist.*

Sir: — In your issue of July 1900 appear three statements with which I should like to deal. The first is of minor importance. Mr. Stead, in his interesting and valuable paper on "Practical Metallography," states that I do not use mechanical means for polishing sections. This is an error. In the micrographic laboratory of the Sheffield University College there is a battery of six 4-inch blocks running by electric power at adjustable speeds. If you care to publish the details of this installation I shall be glad to send you a drawing.

With reference to Mr. Osmond's statement that some metallographists wish to retain the word *crystal* as opposed to *grain* on a "pretext," may I say that personally I use the former term because I consider the so-called grains of metal are usually identical with the mineral allotrimorphic crystals of Rosenbusch, which although they are seldom or never externally geometrical have yet that constant internal orientation which entitles the individual to rank as a crystal, and so distinguishes it from a grain which is internally amorphous. This classification, I think, contains more "logic" than one which assigns to particles of both types of matter the same name. I understand that my friend and colleague, Mr. McWilliam, who is an expert in the crystallography of minerals, will deal more fully with Mr. Osmond's proposed classification.

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The third matter to which I wish to refer is the editorial note in which you state that a photograph by Mr. Lau (which you reproduce) further refutes my statements as to the structure of martensite. The case stands thus. In May 1895 Osmond published his fine treatise "Méthode Générale pour L'Analyse Micrographique de Acier au Carbone." In that he exhibited a photograph of the structure of iron containing 0.45 per cent carbon and 0.46 per cent of impurities (chiefly manganese) quenched at a temperature of 1225° C. This structure he called martensite and stated that all steels containing from about 0.1 to about 1.25 per cent carbon presented a similar appearance when hardened from a full red heat. His photograph is indistinct and at a magnification of 800 diameters shows faintly on a pale grey ground slightly darker interlaced needles, some of which appear to extend across a quarter of the field (Fig. 224, Plate 3).

Arnold, in a paper on the Influence of Carbon on Iron printed in April 1895 and read before the Institute of Civil Engineers in the December of the same year, describing the structure of a hardened steel containing 0.89 per cent of carbon and 0.19 per cent of impurities, stated that the section assumed on etching a "blackleaded" appearance owing to a faint deposit of dark carbonaceous matter. The structure at 600 diameters seemed almost homogeneous and apparently non-crystalline, but probably consisted of minute crystals, the junction lines of which were beyond the range of microscopic vision or were obscured by the faint carbonaceous deposit.

Sauveur, in an admirable paper on the "Micro-Structure of Steel and the Current Theories of Hardening," read in September 1896 before the American Institute of Mining Engineers, stated with reference to the micrographic features of pearlyte and martensite that the latter can never be confused with lamellar pearlyte, but that with granular pearlyte the differentiation was more difficult and the structure must be closely examined. The author then mentioned Osmond's needle modification of martensite. Sauveur also stated that a steel containing 0.21 per cent of carbon and 1.6 per cent of impurity when quenched between Ar_2 and Ar_1 had a structure composed of 31 per cent of martensite irregularly distributed through a matrix of structurally free ferrite. He likewise asserted that the same steel when quenched above Ar_2 at about 800° C. contained 100 per cent of martensite, which he diagramatically figured as practically homogeneous.

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In July 1900 is published in *The Metallographist* a photograph of the structure of steel containing 2.4 per cent of carbon (impurities not stated) quenched at its melting-point. This structure, you, Sir, consider, exhibits white patches and veins of cementite and striated regions which you designate martensite.

As the steel (or mild white iron?) would originally contain about 25 per cent of free cementite I hold that the dark matrix of the section consists of martensite holding some cementite in solution and that the needles you call martensite are really also cementite. I have a section of an experimental steel casting containing about 1.7 per cent of carbon which in its unhardened state presents almost exactly the same structure as that figured in Mr. Lau's excellent photograph. Finally, my position is this: that working with every precaution on nearly pure iron and carbon steels quenched say at 950° C. I get with .2 per cent of carbon a fine granular mixture of pale ferrite and a dark constituent which you, Sir, I think, term "dilute martensite"; with .9 per cent of carbon I get a dark section in which after gently removing the etching deposit I can only distinguish at 600 diameters an indefinite granulation. With 1.5 per cent of carbon I get small, dark, faintly granulated cells of martensite, containing some dissolved cementite and enveloped in thin cell walls of free cementite. These appearances are constant, in experiment after experiment. The foregoing facts seem to point to the conclusion that Osmond, working under certain conditions on impure steels, has obtained martensite crystallized in small needles, and that I, working with purer steels under somewhat different conditions, have obtained the martensite described by you as resembling granular pearlyte. I submit, therefore, that the existence of minutely granular martensite disproves the "lithological entity" claimed for the needle form of martensite by Professor Howe. The pure granular martensite which I described in my paper on the "Influence of Carbon on Iron" has a mass hardness of over 7, i.e., it scratches quartz. For many years I have been in the habit of examining blades quenched by professional hardeners belonging to the Sheffield Blade-makers' Union and had never noted in such blades the needle form of martensite.

Yours faithfully, J. O. ARNOLD.

[The Editor will reply to Professor Arnold's letter in the next number of *The Metallographist.*]

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INDEX.

[The names of authors are printed in SMALL CAPITALS and the titles of articles in *italics*.]

PAGE

Allotropy of Iron 31
Allotropic Theory 52, 165, 248
Alloys, Bearing, Microstructure and Properties of 4
  Constitution of 290
  Law of Phases Applied to 290
  Lead-Bismuth, Microstructure of 128
  Lead-Tin, Microstructure of 127
ARNOLD, J. O., *The Internal Architecture of Steel* 267

BEHRENS, H., and BAUCKE, H., *On Antimonial Alloys for Axle Bearings* 4

Cadmium, Microstructure of 101
CAMPBELL, E. D., *The Constitution of Steel* 13
Carbon, Diffusion in Steel of 16
CARTAUD, G., and OSMOND, F., *New Reagents for the Micrographic Study of Carburized Irons* 1
Cast Iron, Constitution of 290, 293, 300
Critical Points of Iron and Steel, Retardations, Transformations, Recalescence 38, 48, 88, 135
Critical Points, Abnormal Variations of Length Occurring at . . 162

DUMAS, L., *On the Position of the Points of Magnetic Transformation of Nickel Steels* 48

Etching of Polished Sections of Metals and Alloys . . . 1, 78, 230, 319
EWING, J. A., and ROSENHAIN, WALTER, *The Crystalline Structure of Metals* 94

Graphite, Formation in Steel of 30

HOWE, H. M., *The Color-Names for High Temperatures* 43

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PAGE

Iron, Allotropy of 31, 330
Crystallography of 181, 275
Hysteresis of 176
Magnetic Transformations of 39
Microstructure of 98

JOB, ROBERT, *Some Causes of Excessive Heating in Bearing Metals* . 145

LE CHATELIER, H., *Application of the Law of Phases to Alloys and to Rocks* 290
On the Changes of State in Iron and Steel 38
The Dilatation of Iron and Steel at High Temperature 152

Magnetic Transformation of Iron 39
Magnetic Transformation of Nickel Steel 48
Magnetic Properties of Steel Sheets 157
Magnets, Requirements of Steel for 35
Martensite, Structure of 338
Martens, Adolf, Biographical Sketch of 177
Metallography, in the Technical Schools 250
Microscope Accessories for 234
Technology of 220
Value of 83, 86, 87, 176, 331
Metals, Crystalline Structure of 94, 174
For Bearings, Excessive Heating of 145
Strained, Microstructure of 105
MORSE, R. G., *The Effect of Heat-Treatment upon the Physical Properties and the Microstructure of Medium-Carbon Steel* . . . 130

National Physical Laboratory 85
Nickel Steels, Magnetic Transformation of 48

OSMOND, F., *Crystallography of Iron* 181, 275
Steels for Magnets 35
—— and CARTAUD, G., *New Reagents for the Micrographic Study of Carburized Iron* 1
Osmond, F., Biographical Sketch of 255

Photomicrography of Metallic Sections 80, 239
Phosphides, in Steel, Diffusion of 15
Pig Irons, Coke and Charcoal, Microstructure of 154, 165
Phosphorettic, Microstructure of 261
Polishing of Metallic Sections for Microscopical Examination . 77, 95, 223, 318

ROOZBOOM, BAKHUIS, *Iron and Steel from the Point of View of the "Phase-Doctrine"* 293

Digitized by Google

PAGE

ROSENHAIN, WALTER, and EWING, J. A., *The Crystalline Structure of Metals* 94

SAUVEUR, ALBERT, *The Microstructure of Coke and Charcoal Pig Irons* 154
Sorby, Henry Clifton, Biographical Sketch of 89
STANSFIELD, A., *The Present Position of the Solution Theory of Carburized Iron* 24, 300
STEAD, J. E., *Note on the Structure of Phosphorettic Pig Metals* . . 261
Practical Metallography 220
Steel, at Different Temperatures, colors of 41, 43
Constitution of 13, 24, 267, 290, 293, 300
Critical Points of (see Critical Points of Iron and Steel).
Diffusion of Carbon in 16
Diffusion of Phosphides in 15
Diffusion of Sulphides in 13
Dilatation of 152
Effect of Heat-Treatment upon the Physical Properties and the Microstructure of 130
Ferro-Carbons in 17
For Magnets, Requirements of 35
For Rails, Chemistry of 173
Formation of Carbide of Iron in 29
Formation of Graphite in 30
Hardened, Strains in 163
Heat-Treatment of 168, 173
Microscopic Analysis of 64
Microstructure of 52, 134, 168, 244, 267
Modern Theories on the Cause of Hardening of 165
Physics of 330
Solidification of 25
Solution Theory of 24, 300
Steel Shafts, Mysterious Fractures of 320
Steel Sheets, Effect of Annealing on the Magnetic Properties of . . 157
STINE, W. M., *The Microscopic Study of Metals* 314
Sulphides, in Steel, Diffusion of 13

TAYLOR, F. W., and WHITE, MAUNSEL, *Colors of Heated Steel Corresponding to Different Degrees of Temperatures* 41
Tin, Microstructure of 104

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